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### MANUFACTURING PROCESS DEVELOPMENT FOR HIGH-STEENGTH STEELS

TECHNICAL REPORT AFML-TR-66-340

December, 1966

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Manufacturing Technology Laboratory Systems Engineering Group (RTD) Air Force Systems Command Wright-Patterson Air Force Base, Ohio

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## MANUFACTURING PROCESS DEVELOPMENT FOR HIGH-STRENGTH STEELS

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### FOREWORD

This Final Technical Report covers the work performed under Contract \$\textit{A}^33(657)-11277\$ from 1 July 1963 through 30 June 1966. This manuscript was released by the authors in August 1966 for publication as an AFAL Technical Report.

This contract with the Metallurgical Department, Central Alloy District, Republic Steel Corporation was initiated under Manufacturing Methods Project 8-157, "Manufacturing Methods for Maraging Steels". It was ac applished under the technical direction of Mr. George W. Trickett of the Metallurgical Processing Branch (MaIB) Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

The testing program and statistical evaluation were accomplished under the auspices and technical direction of the Metallurgical Department, Messrs. J. T. Shimmin as Project Manager, B. M. Glasgal as Project Engineer, and Mr. J. A. Odar as assistant Project Engineer. Contract Administration and Government lisison was under the direction of Messrs. J. T. Shimmin, Project Manager, George K. Manning, Dr. C. H. Lorig, Francis W. Boulger, Carl T. Olofson, and Arnold F. Gerds of the Department of Process and Physical Metallurgy of Battelle Memorial Institute.

The project was accomplished as a part of the air Force Manufacturing Methods Program, the primary objective of which is to develop on a timely basis, manufacturing processes, techniques, and equipment for use in economical production of USAF materials and components. The program encompasses the following technical areas:

Metallurgy - Rolling, Forging, Extruding, Casting, Fiber, Powder.

Chemical - Propellant, Costing, Ceramics, Graphite, Nonmetallics.

Electronic - Solid State, Materials and Special Techniques, Thermionics.

Fabrication - Forming, Material Removal, Joining, and Components.

Suggestions concerning additional hanufacturing Methods development required on this or other subjects will be appreciated.

This technical report has been reviewed and is approved.

Melvin E. Fields

Colonel, USAF

Chief, Hanufacturing Technology Division

### ABSTRACT

This document constitutes the Final Report on manufacturing process development of 18% nickel maraging steels and 9% nickel. 4% cobalt quench and temper steels. The effect of a number of processing and fabrication procedures on mechanical properties including toughness and stress corrosion was evaluated and is reported herein. None of the grades tested exhibited exceptional sensitivity to forging conditions but there was indication that the 18% nickel maraging steel possessed superior mechanical properties when reduced about 75% and finished at 1800-1850 F. Optimum results were obtained when the 9% nickel-4% cobalt steel with 0.45% carbon was reduced 75% but the 9% nickel-4% cobalt steel with 0.25% carl on exhibited better properties when reduced only 25%. The preferred finishing temperature for these steels was found to be 1900-1950 F. The results also suggest that in most cases vacuum-arc remelting results in some increase in fracture toughness. All of the steels proved to be rather insensitive to the rolling temperature but the greater reduction that the thinner plate underwent apparently resulted in improved toughness at least in the longitudinal direction. Weldability tests indicated that the 18% nickel maraging steel could be welded without severe degradation in heat-affected-zone mechanical properties provided the material was re-aged after welding. Welds of near 100% efficiency were produced in the 9% nickel-4% cobalt steels by quenching and tempering after welding.

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### TABLE OF CONTENTS

														Page
INTRODUCTION		•		•			•	•		•				1
SUMMARY				•		•	٠						•	1
Materials and Ingot Processing	g	•												1
Forgeability	_													2
Rolled-Ring Forgings														3
Closed-Die Forgings														4
Ingots, Slabs, and Heavy Flate								_						4
Sheet and Plate		•							_					4
18Ni Steels								-	_	-	-	-		5
9Ni-4Co Steels				•			•	•	•	•	•	•	•	5
Welding				•	•	•	•	•	•	•	•	•	•	6
werding	• •	•	• •	•	•	•	•	•	•	•	•	•	•	U
STEEL SPECIFICATIONS		•	•		•	•	•	•	•	•	•	•	•	6
Specification RSC-AF-1			. ,		. •	•						4	•	7
Mechanical Properties							•			•			•	7
Specification RSC-AF-2a												•		8
Specification RSC-AF-3a													•	9
Other Qualifying Tests		•	•		•	•	•	•	•	•	•	•	•	11
ABBREVIATIONS		•	•		•	•		•	•		•		•	11
HEATS PRODUCED FOR THE PROC	GRAM	•	•		•	•	•	•	•	•	•	•	•	11
Sources of the Materials		_						_						15
Melting Procedures								_		_	_	-		16
Seventy-Ton Electric-A								-	_	_	_		-	16
Vacuum-Arc Remelting									•	•	•	•	•	17
Electroslag Remelting P						•		•	•		:	-	•	18
QUALIFYING-TEST RESULTS					•				•	•				19
INGOT FORGING		•	•		•	•	•	•	•	•	•	•	•	19
i8Ni-Steel			•		•		•	•	•	•	•	•	•	19
9Ni-4Co Steel					•		•	•	•	•		•	•	21
Flame-Cutting Experiments or	n 18N	i Bil	lets		•	•	•	•	•	•	•	•	•	22
FORGEABILITY EVALUATIONS .						•		٠	-	÷	•	•		22
Forging Finishing-Temperatu	re Stu	ıdv					_			_	_			22
Macroscopic and Micros							-		_	•	-	-		23
Tensile-Test Properties							•	:	•	•	•	•	•	24
Fracture-Toughness Te			•	•	•		•	•	•	•	•	•		24
Finishing Temperatures				• •			•	•	•	•	•	•	•	25
rinishing remperatures	•	•	•	•	•	•	•	•	•	•	•	•	•	

### ESTEVIOUS PAGE WAS BLANK, THE REPORT WAS NOT FILMED.

# TABLE OF CONTENTS (Continued)

														Page
Forging-Reduction Studies	•					,								25
Materials														25
Tensile Properties														26
Fracture-Toughness Properti	es													31
Impact Properties														33
Metallographic Examination .	•													35
Conclusions From Forging Re														<b>3</b> 6
EVALUATION OF ROLLED-RING FORGI	NGS (	OF	18	Ni(	25(	)) S	TE	EL			•		•	37
Materials														37
Materials Acceptance Tests		•	•	•	•	•	•	•	•	•	•	•	•	37
														38
Forging Procedure														41
Mcchanical Properties of Roll														
Statistical Analyses Procedur														47
Results of Statistical Analysis	3.	•	•	• .	•	•	•	•	•	•	•	•	•	48
EVALUATION OF RING-ROLLED FORGI	NGS	OF	18	Ni(	(300	)) <i>F</i>	NI	9	Ni-	4C	0			
STEEL	•	•	•	•	•	•	•	•	•	•	•	•	•	62
Prior History										 •				62
Tensile Properties														64
Fracture Toughness		•						_	_	_	_	_		66
Metallographic Examinations				•		•	•	•	· c	•		•	•	67
EVALUATION OF CLOSED-DIE FORGING	GS.							•	•	•	•	•	•	68
Material														68
Machining and Heat Treatment of Sp														71
Tersile Tests														71
Chirpy Tests	• •	•	•	•	•	•	•	•	•	•	•	•	•	75
Charpy Tests	• •	•	•	•	•	•	•	•	•	•	•	0	•	75
Fatigue			•	•	•	•	•	•	•	•	•	•	•	77
Susceptibility to Stress-Corrosion	Crac.	Kin	3	•	•	•	•	•	•	•	•	•	•	
Fracture Toughness	• •	•	•	•	•	•	٠	•	•	•	•	•	•	80
EVALUATION OF INGOTS, SLABS, AND	4-IN	ICH	P	LA	ΤE	•	•	•	•	•	•	•	•	85
Tensile and Fracture-Toughness To	ests												•	85
Metallographic Examinations .						•		c	•	•	•	•	•	93
PROCESSING AND EVALUATION OF SHI	EET .	AN:	D I	PL/	ATI	€.		•	٠	•	٠	•	•	94
Processing Studies on 18Ni Sheet a	nd 101	ato												94
							•	•	•	•	•	•	•	94
Melting and Fabrication Back							•	•	•	•	•	•	•	95
Testing Conditions								•	•	•	•	•	•	
Hot-Rolling Studies - 3/8-Inc	ch Pi	31 E		•	•			•	•	•				95

# TABLE OF CONTENTS (Continued)

							Page
Hot Rolling Studies - 0.160-Inch Sheet							95
Gross-Rolling Studies							99
Cross Rolling							ųq
Heat-Treatment Studies							မှုတဲ့
Processing Studies of 9N1-4Co Sheet and Plate							107
Melting and Fabrication History							107
Testing Conditions							108
Hot-Rolling Studies - 3/8-Inch Plate							108
Hot-Rolling Studies - 0. 160-Inch Sheet							108
Cross-Rolling Studies							112
Heat Treatment Studies - 3/8, 3/4-Inch Plate							114
Heat-Treatment Studies - 0.160-Inch Sheet							114
							117
Martensitic Specimens	•	•	•	•	•	•	117
Bainitic Specimens							120
Evaluation of 18Ni Steel Plate and Sheet							
Prior History							120
Tensile Properties							120
Charpy Properties							124
Precracked-Charpy-Impact Properties							125
Environmental Effects							125
Fracture Toughness							128
Metallographic Examinations	•	•	•	•	•	•	135
Evaluation of 9Ni-4Co Steel Plate and Sheet							136
Prior History							136
Tensile Properties							137
Charpy Properties							137
Precracked Charpy Impact Properties	•			•	•	•	139
Environmental Effects	•					•	139
Fracture Toughness	•						141
Metallographic Examinations					•	•	142
WELDING EVALUATION				•		•	143
Objectives	•						143
Materials							143
Simulated Heat-Affected Zone Studies					۵		145
Hot Ductility Tests				•			145
Room-Temperature Tests on Simulated HAZ Speci							145
9Ni~4Go Steels		4			2		147
Transweld Properties of TIG Weldments							149
18Ni Steels							150
9Ni-4Co Steels							156
	-	•		-			
CONCLUSIONS AND RECOMMENDATIONS	•	•	•	•	•	•	162
18Ni Maraging Steel							162
9Ni-4Co Steel	•					•	162
Free two contracts of the first terms of the first		-		-			

### LIST OF ILLUSTRATIONS

Figu		Page
1	Fracture-Toughness Specimen	31
2	Load-Deflection Gurves of 18Ni-(250) Fracture-Toughness Specimens Exhibiting Pop-In Beyond Proportional Limit	34
3	Strength of 18Ni Steel as Influenced by Solution-Annealing Temperature	50
4	Strength of 18Ni Steel as Influenced by Melting Process	51
5	Ductility of 18Ni Steel as Influenced by Testing Direction	52
6	Fracture Toughness of 18Ni Steel as Influenced by Testing Direction	53
7	Fracture Toughness of 18Ni Steel as influenced by Testing Direction and Melting Process	55
8	Fracture Toughness of 18Ni Steel as Influenced by Forging Practice.	56
9	Fracture Toughness of 18Ni Steel as Influenced by Rolling Temperature	57
10	Reduction of Area Values Per Circumferential Specimens of 18Ni Steel as Influenced by Rolling Temperature	58
11	Circumferential Fracture Toughness as Influenced by the Amount of Wall Reduction at Final Rolling	60
12	Circumferential Ductility of 18Ni Steel as Influenced by the Amount of Wall Reduction at Final Rolling	61
13	Locations of Specimens Used for Evaluating Properties of Ring-Rolled Forgings	63
14	Location of Test Specimens in Forgings	70
15	Tensile-Test Specimens	74
16	Notched Axial Fatigue Specimens	76
17	Notched-Axial-Fatigue Properties of High-Strength Steel Forgings .	78
18	Low-Cycle - High-Stress Notched-Axial-Fatigue Properties of High-Strength Steel Forgings	79
19	Stress-Corrosion Specimen	80

# LIST OF ILLUSTRATIONS (Continued)

Figure		Page
20	Center-Notch Tensile Specimen for Evaluating the 0.045 and 0.160-Inch-Thick Sheet Material	96
21	The Effect of Rolling Temperature on the Mechanical and Fracture- Toughness Properties of 3/8-Inch Plate in Heat-Treated 18Ni(250) VAR, Heat 3920864	97
	VAR, Heat 3720004	<i>,</i> ,
22	The Effect of Cross-Rolling on the Mechanical and Fracture- Toughness Properties of 3/8-Inch Plate in Heat-Treated 18Ni(250) VAR, Heat 3940864	100
23	The Effect of Cross Rolling on the Mechanical and Fracture- Toughness Properties of 0.170-Inch Sheet in Heat-Treated 18Ni(250)	101
	VAR, Heat 3920864	101
24	The Effect of Solution-Annealing Temperature and Re-Solution Annealing on the Mechanical and Fracture-Toughness Properties of 3/8-Inch Plate in Heat-Treated 18Ni(250) VAR, Heat 3920864	103
25	The Effect of Solution-Annealing Temperature and Re-Solution Annealing on the Mechanical and Fracture-Toughness Properties of 0.160-Inch Sheet in Heat-Treated 18Ni(250) VAR, Heat 3920364	104
26	The Effect of Solution Annealing and Aging Temperatures on the Mechanical and Fracture-Toughness Properties of 3/8-Inch Plate in Heat-Treated 18Ni(250) VAR, Heat 3920864	105
27	The Effect of Solution Annealing and Aging Temperatures on the Mechanical and Fracture Toughness Properties of 0.160-Inch Sheet in Heat-Treated 18Ni(250) VAR, Heat 3920864	106
28	The Effect of Rolling Temperature on the Mechanical and Fracture-Toughness Properties of 3/8-Inch Plate in Heat-Treated 9Ni-4Co(200) AM-Si/Al-DOX, Heat E5565	109
29	The Effect of Rolling Temperature on the Mechanical and Fracture-Toughness Properties of 3/8-Inch Plate in Heat-Treated 9Ni-4Co(200) VAR-C-DOX, Heat 3920795	110
30	The Effect of Cross Rolling on the Mechanical and Fracture- Toughness Properties of 3/8-Inch Plate in Heat-Treated 9Ni-4Co(200) VAR-C-DOX, Heat 3920795	113
31	The Effect of Tempering Temperature and Temper Mode on the Mechanical and Fracture-Toughness Properties of 3/8-Inch Plate in Heat-Treated 9Ni-4Go(200) VAR-G-DOX, Heat 3920795	115

# LIST OF ILLUSTRATIONS (Continued)

Figure		Page
32	The Effect of Tempering Temperature on the Mechanical and Fracture Toughness Properties of 0.160-Inch Sheet in Heat-Treated 9Ni-4Go(250)VAR-C-DOX, Heat 3920852	118
3 3	The Effect of Transformation Temperature and Time on the Mechanical and Fracture-Toughness Properties of 0. 160-Inch Sheet in Heat-Treated 9Ni-4Co(250)VAR-C-DOX, Heat 3920852	119
34	The Relationship of Strength to Fracture Toughness in Bainitic and Martensitic Microstructures of 0.160-Inch Sheet in Heat-Treated 9Ni-4Co(250)Si/Al-DOX-VAR, Heat 3920851	121
35	The Relationship of Strength to Fracture Toughness in Bainitic and Martensitic Microstructures of 0. 160-Inch Gage Sheet in Heat-Treated 9Ni-4Co(250)VAR-C-DOX, Heat 3920852	122
36	Environmental Test Specimen for Evaluating 0.045 and 0.160-Inch-	
	Thick Sheet Material	126
37	Part-Through-Crack (PTC) Tensile Specimen for Evaluating 0. 160-Inch-Thick Sheet Material	130
38	Part-Through-Crack (PTC) Tensile-Specimen Configuration for Evaluating the 3/8-Inch-Thick Plate Materials	131
39	Part-Through-Crack (PTC) Tensile-Specimen Configuration for Evaluating the 3/4-Inch-Thick Plate Materials	132
40	Notch Tensile Specimen for Evaluating 3.0-Inch-Thick Flate Materials	134
41	Weld-Joint Designs and Welding Parameters	151
42	Face- and Side-Bend and Transweld-Tensile-Specimen Configurations	152
43	Partial Thickness Fracture Toughness Specimen	153
	LIST OF TABLES	
Table		
1	Summary of the General Processing Procedures Produced for the Various Heats Employed in the Program	12
2	Chemical Analyses of the Steels	13

# LIST OF TABLES (Continued)

Table		Page
3	Tensile-Properties Heat-Qualification Tests	20
4	Typical Rockwell C Hardnesses of the 9Ni-4Co and 18Ni-Co-Mo Steels in the Heat-Treated Condition	27
5	Average Mechanical Properties of 9Ni-4Co and 18Ni Steels as Determined in Room-Temperature Tests	28
6	Relative Merit of Melt Practice at Optimum Forging Reduction Based on Optimum Air-Melt Properties for 18Ni Maraging and 9Ni- 4Co Quench and Temper Steels	30
7	Calibration-Equation Coefficients for 1/2 x 1/2-Inch Notch-Bend Fracture-Toug'mess Specimens - Three Point Loading	32
8	Material-Acceptance Mechanical-Property Test Results for 18Ni(250) Maraging Steels	39
9	Details of Forging Program for Producing Two Diameters of Seamless Rings From 18Ni(250) Maraging Steels at Ladish	40
10	Material Requirements and Scheduled Forging and Seamless-Ring-Rolling Procedures	41
11	Yield-Strength Data for 18Ni Steel	42
12	Ultimate-Tensile-Strength Data for 18Ni Steel	43
13	Percent Elongation Test Data for 18Ni Steel	44
14	Percent-Reduction-of-Area Test Data for 18Ni Steel	45
15	Precracked Charpy V-Notched Impact Data for 18Ni Steel	46
16	Melting and Processing History of Ring-Rolled Forgings	62
17	Chemical Compositions of Ring-Rolled Forgings	64
18	Mechanical Properties of Specimens From Rolled-Ring Forgings	65
19	Precracked Charpy Impact Values for 9Ni-4Co(200) Samples From Ring-Rolled Forgings	67
20	Chemical Composition of Steel Forgings	69
21	Tensile Properties of I8Ni(300) Vacuum Arc Remelted Steel Forging Code "K"	72

# LIST OF TABLES (Continued)

Table		Page
22	Tensile Properties of Forgings Heat Treated to Ultimate Strengths Ranging From 260 to 280 KSI	73
23	Charpy V-Notch Values of 18Ni and 9Ni-4Co Steels	<b>7</b> 5
24	Stress-Corrosion Test Data on Round, Unnotched Tensile Specimens With Short-Traverse Grains Exposed	82
25	Fracture-Toughness Properties of Specimens Taken From Experimental Forgings	83
26	Ranges in Chemical Compositions and Mechanical Properties of 18Ni(250) Ingots	86
27	Tensile and Fracture-Toughness Values for Specimens Taken From 10 x 40-Inch Slabs of 18Ni(250) Steel	87
28	Tensile and Fracture-Toughness Plate Values for Samples Taken From 4-Inch Plates of 18Ni(250) Steel	90
29	Fackground Information on 18Ni Maraging-Steel Heats Used for Rolling and Heat-Treatment Studies	94
30	Effect of Rolling Temperature on Properties of 0.160-Inch-Thick Sheet of 18Ni Steel	9٤
31	Background Information on 9Ni-4Co Alloy Steels Used for Rolling and Heat-Treatment Studies	107
33	Effect of Rolling Temperature on Properties of 0. 160-Inch-Thick Sheet of 9Ni=4Co (250) Steel	111
33	Response of Samples From 3/4-Inch Plates of 9Ni-4Co(200) VAR-C-DOX Alloy Steel (Heat No. 3920795) to Various Tempering Treatments.	116
34	Prior History of Plate and Sheet Products Used for Evaluation of 18Ni Steels	120
35	Tensile Properties of Heat-Treated 18Ni Sheet and Plate	123
36	Tensile Properties of Heat Treated 18Ni(200) 0,65-Inch Steel Plate	124
37	Charpy Impact Properties of Heat-Treated 18Ni Plate	125
38	Precracked Charpy Impact Properties of 18Ni Steels	125
39	Failure Times for Fatigue-Precracked Tensile Specimen of 18Ni Steels Immersed in 3 Percent NaCl at 80 K	127

# (Continued)

Table		Page
40	Fracture-Toughness Values, K <sub>C</sub> , for Longitudinal and Transverse Specimens of 18Ni Steel	129
41	Fracture-Toughness Values, K <sub>IC</sub> , for Longitudinal and Transverse Specimens of 18Ni Steel	133
42	Prior History of Plate and Sheet Products Used for Evaluation of 9Ni-4Co Steels	135
43	Tensile Properties of Heat Treated 9Ni-4Co(250) Sheet	137
44	Tensile Properties of Heat Treated 9Ni-4Co(200) Plate	138
45	Charpy Impact Properties of Heat-Treated 9Ni-4Co(200) Plate	139
46	Failure Times for Fatigue-Precracked Tensile Specimens of 9Ni~4Co Steels Immersed in 3 Percent NaCl Solution at 80 F	140
41	Fracture Toughness Properties of Heat Treated 9Ni-4Co(250) Sheet	141
48	Fracture-Toughness Values, K <sub>IC</sub> , for Longitudinal and Transverse Specimens of 9Ni-4Co Steel	142
49	Tensile Tests on Parent-Metal Specimens	154
50	Tensile Results of Welded Specimens	154
51	Guided-Face Bend-Test Results for 0.160-Inch-Thick Material	155
,52	Guided-Face Bend-Test Results for 0.375-In. Thick Material	155
53	Guided Side Bend Test Results for 0.750-Inch-Thick Material	155
54	Transweld PTC Fracture-Toughness Test Results for 1811 Steel	157
55	Tensile Results of Parent-Metal Specimens	157
56	Tensile Results of Welded Specimens	158
57	Guided-Face Bend-Test Results for 0, 160-Inch-Thick Material	159
58	Guided-Face Bend Test Results for 0.375-Inch-Thick Material	159
59	Guided-Side Bend-Test Results for 0.750-Inch-Thick Material	159
60	Transweld PTC Fracture-Toughness-Test Results for 9Ni-4Co Steel .	161

### INTRODUCTION

The investigations describes in this report were concerned with the manufacturing development of high-strength steels and were intended to determine the best manufacturing and processing procedures for the production of 18Ni maraging and 9Ni-4Co quench and temper alloy-steel forgings and flat-rolled product. The Air Force initiated the program to develop the optimum manufacturing methods for the production of the two alloy steels as forgings, plate, and sheet and to partially assess the degree of uniformity and reproducibility that could be expected in mechanical properties. The major subcontractors that participated in the program were:

Aerojet-General Corporation, Sacramento, California

Ladish Company, Cudahy, Wisconsin

Wright Aeronautical Division, Curtiss-Wright Corporation, Wood-Ridge, New Jersey

General Dynamics/Fort Worth, Texas

North American Aviation, Inc., Los Angeles Division, Los Angeles, California.

Because of the rapid advance of hardware-design technology, particularly in the aircraft and aerospace industries, an urgent demand has developed for more reliable, very high-strength construction materials. A concerted effort by material engineers to meet the design engineer's demands has resulted in significant improvement in the reliability of high-strength materials. Major interest developed in two alloy steels, one strengthened by "age hardening" and the other strengthened by the more conventional "quench and temper" treatment. The two steels are the 18Ni maraging steel introduced by the International Nickel Company and the 9Ni-4Co quench and temper steel developed by Republic Steel Corporation. The 18Ni steel was studied at a nominal strength of 250,000 and 300,000 psi and the 9Ni-4Co steel at a nominal strength of 200,000 and 250,000 psi.

The program called for evaluation of the 18Ni and 9Ni-4Co alloy steels as produced by three melting practices for each grade. Variations in production procedures for open-die forging, in the production of large-diameter ring forgings of the 250,000-psi maraging steel, and in hot rolling of sheet and plate were studied for their effects on metallographic structure and mechanical properties, particularly fracture toughness.

### SUMMARY

### Materials and Ingot Processing

As the first phase of the study, an extensive literature survey of 18Ni-maraging and 9Ni-4Co-quench and temper steels was conducted. It was decided that material in the air-melted, vacuum-degassed, and vacuum-arc-remelted condition should be

studied. Two different compositions of 18Ni-maraging steel were included: one capable of achieving about 250,000 psi strength and the other capable of reaching a strength of nearly 300,000 psi. Two different compositions of the 9Ni-4Co steel were also included. One was specified 0.24 to 0.30 percent carbon and was expected to exhibit a strength of nearly 200,000 psi, and the other was specified 0.42 to 0.48 percent carbon and was expected to exhibit a strength of about 250,000 psi. The literature survey permitted the establishment of tentative specifications for materials to be used in the program that set limits on chemical composition, mechanical properties and cleanliness, and also described the thermal cycles to be employed for forging and for heat treatment. Heats were qualified and accepted for the program by forging 5-inch-square bars from samples cut from 8 x 16-inch blooms in most cases.

Most of the steel used in the program was produced by Republic Steel Corporation, but several heats of both the 18Ni and the 9Ni-4Co grades were made by other steel companies. This permitted some comparison of material produced at different mills, and it also contributed to more widespread know-how in producing these high-strength steels.

No difficulty was encountered in forging the 18Ni steels until an attempt was made to reduce two 43-inch octagon section air melt ingots to 30-inch round cornered squares. Both of these cracked. The forging practice was changed to a higher temperature (2300 F) and longer soak, to a slower cooling rate from 1200 F to 200 F after forging, and to include a postforging treatment of 300 F to 500 F for 36 hours. After making these changes, no further difficulty was encountered in forging the 18Ni steels. Cracks were formed during the initial breakdown of several of the first 9Ni-4Co ingots that were forged. This difficulty was overcome by careful surface preparation prior to heating for forging and by increasing the initial temperature from 2050 F to 2200 F.

It was observed that samples flame-cut from blooms and billets of 18Ni steel in some cases developed cracks that extended across the flame-cut surface. Tests demonstrated that such cracking could be avoided if the blooms or billets were heated to 1100 F (overaged) and cooled to room temperature prior to flame cutting, or if the blooms or billets were flame cut while cooling from 1500 F. Flame cutting while the steel was at several hundred degrees presented practical difficulties, and the prior treatment was considered to be the more practical solution.

### Forgeability

The forgeability of the grades of steel involved in the program was evaluated with respect to finishing temperature and degree of reduction. Four-inch-square by five-inch specimens were upset to 1-inch thickness in a single stroke by a 500-ton press for the temperature study. The pancake specimens were examined as forged, then heat treated and used for tensile specimens and precracked Charpy specimens. All of the steels were considered to be forgeable. It was concluded on the basis of mechanical properties after heat treating that the most desirable finishing temperature for the 18Ni air-melted and vacuum-degassed steels was 1900 to 1950 F; that the most desirable finishing temperature for the 18Ni vacuum-arc-remelted steel was 1850 to 1900 F; and that the most desirable finishing temperature for the 9Ni-4Co steels (all types) was 1900 to 1950 F.

The fracture-toughness tests indicated that finishing temperature had little or no effect on the final toughness and that the vacuum-arc-remelted, carbon dioxidized 9Ni-4Co steel possessed toughness superior to that of the air-melted aluminum deoxidized 9Ni-4Co steel. The superior toughness of vacuum-arc-remelted material was frequently observed in subsequent tests.

The forging reduction portion of the study involved reducing 8-inch-thick billets to specimens that were 2, 4, and 6 inches in thickness, which represented reductions of 75, 50, and 25 percent, respectively. It was concluded that optimum processing for each grade was 75 percent reduction for the 18Ni (300), 18Ni (250), and 9Ni-4Co (250) grades, and 25 percent reduction for the 9Ni-4Co (200) grade. Preferred finishing temperatures were 1800 to 1850 F for all 18Ni steels and 1900 to 1950 F for all 9Ni-4Co steels.

### Rolled-Ring Forgings

The influence of melting practice and forging procedures on the structure and mechanical properties of rolled-ring forgings of 18Ni (250) steel was studied by the Ladish Company. Four rings of air-melted steel and four rings of vacuum-arc-remelted steel were produced. The rings represented two different degrees of wall reduction (75 and 50 percent) and two different forging temperatures (1950 and 2250 F). In addition, the test specimens were solution annealed at two different temperatures, 1650 F and 1500 F.

Statistical analysis of the data obtained indicate that the vacuum-arc-remelted material had about 3000-psi-higher yield and ultimate strengths and at the same time about a 10 percent-higher precracked-Charpy value than the air-melted material. "Circumferential specimens" (specimens cut with their long axes in the circumferential direction) exhibited greater elongation, reduction of area, and precracked Charpy values than "transverse specimens". Both precracked-Charpy values and reduction-of-area values suggest that it is advantageous to heat to 2250 F and forge while the temperature falls to 1950 F, rather than heating to 1950 F and reheating frequently. Greater precracked-Charpy values, elongation, and reduction of area were exhibited by circumferential specimens that had received 75 percent reduction than by specimens that had received 50 percent reduction. The 1500 F solution anneal appeared to result in slightly ligher strength and lower ductility than the 1650 F solution anneal. These results suggest that the preferred processing should be:

- (1) Vacuum-arc remelting
- (2) Heating to 2250 F before forging, and forging while he temperature drops to 1950 F.

Other rings made of 18Ni (300) and 9Ni-4Co (250) and 9Ni-4Co (200) steel were evaluated by Aerojet-General Corporation. The results indicate the 9Ni-4Co (200) steel to be, by a considerable margin, the toughest of the three grades, with the 9Ni-4Co (250) of intermediate toughness and the 18Ni (300) of lowest toughness. Thus, it was apparent that there is an inverse relationship between strength and toughness, as has been shown on previous occasions. The grade of steel selected for a given part will depend upon the relative importance assigned to strength and ductility.

### Closed-Die Forgings

Forgings weighing 38 pounds each were produced in the dies used for the Wing Pivot Forward Support Bracket for the F-111 aircraft. Test specimens of various kinds were cut from the forgings. The materials employed were 18Ni (300) vacuum-arc-remelted; 18Ni (250), both air melted and vacuum-arc-remelted; and 9Ni-4Co (250), vacuum-arc-remelted. Notched and unnotched tensile properties, slow-bend fracture-toughness fatigue strength, and stress-corrosion resistance were evaluated. The results of the tensile tests show a remarkable independence of specimen orientation on elongation and reduction of area as well as tensile and yield strengths for the vacuum-arc-remelted material. The air-melted 18Ni (250) material, however, exhibited elongations and reductions in area that were noticeably lower than its vacuum-melted counterpart. The air-melted material was somewhat higher in both yield and ultimate strengths. Therefore, there is some question about how much of the loss in Juctility can be attributed to the melting practice.

A comparison between the 18Ni (300) vacuum-arc-remelted material and the 18Ni (250) vacuum-arc-remelted material is quite interesting. The difference in both yield and tensile strength was about 30 ksi (290 and 285 ksi versus 260 and 255 ksi). The notch-unnotched ratio was substantially lower for the 18Ni (300) material, even though the decrease in elongation and reduction of area was quite small. The 18Ni (300) possessed lower V-notched Charpy values at both room temperature and -65 F, almost identical notched fatigue strength, greater susceptibility to stress-corrosion cracking, and  $G_{\rm C}$  values lower by a factor of almost 2. Thus, it is apparent that the higher strength of the 18Ni (300) is achieved with a conspicuous loss in ductility and toughness.

The air-melted 18Ni (250) steel, although exhibiting a little higher yield and ultimate strength than the vacuum-arc-remelted 18Ni (250) steel, was inferior to the vacuum-arc-remelted material in other respects, particularly with respect to V-notched Charpy values and notch-unnotched tensile ratio.

### Ingots, Slabs, and Heavy Plate

A study was made of the ten ile properties and fracture toughness of 18Ni (250) vacuum-arc-remelted steel when cast in various-size ingots and reduced to 10-inch-thick slabs and 4-inch-thick plate. The reproducibility of specimens cut from ingots was poor and of doubtful significance, particularly with respect to elongation, reduction of area, and G<sub>IC</sub> values.

The data on specimens cut from slabs and plate reduced from 18, 24, and 32-inch-diameter ingots was not entirely consistent.

### Sheet and Plate

Tensile V-notched Charpy and center-notched fracture-toughness tests were used to evaluate the effect of plate and sheet-rolling temperature, thickness, cross rolling,

and heat treatment. Air-melted and vacuum-arc-remelted 18Ni (250), vacuum-arc-remelted 18Ni (300), and air-melted and vacuum-arc-remelted 9Ni-4Co in both the (200) and (250) classes were included in this portion of the program. Five different thicknesses (3.0, 0.75, 0.375, 0.160, and 0.045 inches) were evaluated.

### 18Nı Ste**e**is

The effect of rolling temperature on the properties of the 18Ni steels was relatively small. There appeared to be some advantage in finishing plate and sheet bar of 18Ni steel at 1950 F and in finishing 18Ni steel sheet at 1550 F. Cross rolling with 67 percent reduction increased the fracture toughness of the 18Ni 3/8-inch plate and 0.160-inch sheet. These latter results were somewhat surprising in view of the lack of anisotropy in the straightaway rolled material. A single solution anneal for 1 hour at 1500 F followed by aging for 3 hours at 925 F seemed to develop the best combination of strength and ductility. However, the difference observed in properties after use of higher solution-annealing temperatures (up to 1650 F) and aging at either 900 F or 950 F was not great.

No consistent effect of melting practice on the tensile properties (including elongation and reduction of area) was observed. Furthermore, tensile properties in the longitudinal and long transverse directions were not influenced by plate thickness. V-notched Charpy and precracked Charpy specimens did not indicate that melting practice or plate thickness had a significant effect. Both  $K_{\rm c}$  and  $K_{\rm Ic}$  were determined. More than half the results were invalid by ASTM standards because the net stress exceeded 80 percent of the yield strength. The results, nevertheless, suggest that for a given yield strength the vacuum-melted steel is superior to the air melted; that the lower the titanium, the greater the fracture toughness, and that 3/8-inch and thinner plate or sheet is superior to heavier material.

Stress-corrosion tests in dilute NaCl solutions, though limited in number, show that the lower the tensile strength, the greater the resistance to de ayed fracture, and that vacuum-arc melting is superior to air-melted material.

### 9Ni-4Co Steels

Both the (250) and (200) grades of these steels proved to be rather insensitive to rolling temperature. The plate and sheet bar of the (200) grade possessed a slightly better combination of strength and toughness when finished at 1950 F. The sheet processed from the (250) grade appeared best when the sheet bar was finished at 1850 F and the sheet was finished at 1500 F. Double tempering at 1000 F for 2 hours each cycle resulted in the most desirable combination of strength and toughness for the (200) material, and austempering at 475 F produced the best properties in the (250) material. The 9Ni-4Co steels were more susceptible to stress corrosion than the 18Ni steels. Crack-growth rates at 80 percent of the yield strength in dilute solution of NaCl were roughly 4000 inicroinches per minute, some 20 times as fast as with the 18Ni steels.

### Welding

Both the 18Ni and the 9Ni-4Co steels were evaluated for weldability by North American Aviation, Incorporated. Tests included determining the susceptibility to hot cracking, the mechanical properties of simulated heat-affected zone material, and the mechanical properties of specimens welded by the tungsten-inert-gas method. The high-temperature strength and ductility of all the grades except the 18Ni (250) air-melted grade followed the expected pattern of abrupt loss in ductility as the melting point was approached and immediate recovery of ductility on cooling from the hot short temperature range. The 18Ni (250) air-melted heat included in this program exhibited erratic ductility until cooled below 2000 F.

The 18Ni steels exhibited a loss in notch strength as well as tensile strength after heating to 1200 F in simulated heat-affected zone tests, apparently because of overaging. The same materials exhibited an even larger (40 percent) loss in notched strength when cycled between 1800 F and 2500 F, apparently as the result of resolution. The loss in strength caused by single-cycle heating above 1800 F was almost completely recovered by reaging. Ten-cycle heating above 1800 F (as might be encountered in some heat-affected zones) resulted in a 10 to 20 percent loss in strength even after reaging.

Precracked Charpy specimens were used to evaluate fracture toughness of simulated heat-affected zone material. Some loss in absorbed energy was experienced after cycling the 18Ni steel to 2500 F and reaging, but the loss was not large, and although probably statistically significant would be classed as a minor effect.

The 9Ni-4Co (250) steel containing 0.45 percent carbon was completely re-heat treated after being subjected to simulated heat-affected zone thermal cycles. It substantially regained its original strength and fracture toughness, and under these conditions was unaffected by the thermal cycles. The 9Ni-4Co (200) steel was tested as cycled and after being reheated to 50 F below the initial tempering temperature. In the as-cycled condition strength increased progressively as the peak temperature was raised from 1300 to 1500 F because of increasing amounts of newly formed martensite. Cycling at still higher temperatures caused increasing brittleness because of increasing grain size without further increase in strength. Tempering at 50 F below the initial tempering temperature lowered the strength and restored much of the toughness.

Tungsten inert-gas-welded specimens of the 18Ni steels possessed strengths equivalent to the original material when tested with the weld bead on. A considerable loss in ductility and toughness was indicated by notched-tensile and slow-bend tests. Welded specimens of the 9Ni-4Co (250) steel tested with the weld bead on broke outside the weld. Welded specimens of the 9Ni-4Co (200), re-heat treated after welding, exhibited strength and ductility equal to the original material.

### STEEL SPECIFICATIONS

The first phase of the program was a very complete literature survey on both the 18Ni and 9Ni-4Co steels. As a result of the survey it was possible to prepare rather complete material specifications for the steels to be used in the program. These

specifications were then used as a basis for qualifying the heats prepared for the program. The qualifying tests, for the most part, were performed on samples taken from 8 by 16-inch billets. The more important aspects of the three specifications employed are indicated below. They were designed to permit use of typical production-quality material, as differentiated from selected and premium-quality material.

### Specification RSC-AF-1

This specification applies to three different classes of maraging steel as indicated below.

Class I, air melt, nominal strength of 250 ksi.

Class II, vacuum-arc remelt, nominal strength of 250 ksi.

Class III, vacuum-arc remelt, nominal strength of 300 ksi.

The specification placed limitations on the chemical composition, mechanical properties, and nonmetallic inclusions, as indicated below.

	Chemical Analysis,	percent
Element	Class I and Class II	Class III
C	0.03 max	0.03 max
Mn	0.10 max	0,10 max
Si	0.10 max	$0.10  \mathrm{max}$
$\mathbf{p}$	0.010 max	0,010 max
S	0.010 max	0,010 max
Ni	17, 5-18, 5	18.0-19.0
Co	7.5-8.5	8.5-9.5
Mo	4.7-5.2	4.7-5.2
Al	0.05-0.15	0,05-0.15
Ti	0,30-0,50 (aim 0,38)	0.60-0.90
в	0.003 added	0, 003 added
Zr	0.02 added	0,02 added
Ca	0,05 added	0.05 added

### Mechanical Properties

Heat Treatment (Solution anneal 1500 F, 1 hour, air cool, age 900 F, 3 hours, air cool).

Mechanical Property	Class I	Class II	Class III
Yield strength, ksi	240 (min)	240 (min)	270 (min)
Tensile strength, ksi	250 (min)	250 (min)	280 (min)
Reduction of area, percent	30 (min)	30 (min)	. 25 (min)
Elongation, percent	6 (min)	6 (min)	5 (min)

Magnetic Particle Inspection.

•	Class I	Class II	Class III
AMS Step-Down Test	2301-A	2300-A	2300-A
Frequency/Severity Rating (F/S)	1, 10/1, 05	0.50/1.00	0.50/1.00

Microcleanliness (J-K Rating to ASTM E45-61).

Inclusion Type		Incl	lusion Rati	ngs (Worst )	Field)		
	Class I		Cla	Class II		Class III	
	Thin	Heavy	Thin	Heavy	Thin	Heavy	
A	2.0	1.5	1.5	1.0	1.5	1.0	
В	2, 0	1.5	1.5	1.0	1.5	1.0	
С	2.0	1,5	1.5	1.0	1.5	1,0	
D	2.0	1.5	2.0	1.5	2.0	1, 5	
E	4.0	2, 5	3.0	2.0	3.0	2.0	

### Specification RSC-AF-2a

In a similar manner, this specification applied to three classes of 9Ni-4Co steel possessing a nominal strength of 200,000 psi. These three classes were:

Class I, air melt, Si/Al-deoxidized steel

Class II, air melt, Si/Al-deoxidized, vacuum-arc-remelt steel

Class III, air melt, vacuum-arc-remelt steel, carbon-deoxidized.

It specified the following chemical composition, mechanical properties, and cleanliness:

Chemical Analysis (All Three Classes).

Element	Percent		
С	0.24-0.30		
Mn	0.10-0.35		
Si	0.20-0.35(a)		
P	0.01 max		
S	0,01 max		
Ni	7, 0-8, 0		
Co	3.5-4.5		
Мо	0.35-0.60		
Cr	0.35-0.60		
V	0,06-0,12		

(a) In carbon-deoxidized steel, 0.10% maximum silicon was specified. Heat I eatment.

malize 1650 F, 1 hour, air cool

Austenitize 1550 F, 1 hour, oil quench

Double temper 2 hours each at 1000 F, air cool.

Mechanical Properties (All Three Classes).

Mechanical Property	
Yield Strength, ksi	175 (min)
Ultimate Tensile Strength, ksi	185 (min)
Reduction of Area, percent	55 (min)

13 (min) Clongation, percent

Magnetic-Particle Inspection.

	Class I	Class II	Class III
AMS Step-Down Test	2301~A	2300-A	2300-A
Frequency/Severity Rating (F/S)	1,00/0.75	0.50/1.00	0,50/1.00

Microcleanliness (J-K Rating to ASTM E45-61).

Inclusion Type		Incl	usion Ratir	ngs (Worst F	`ield)	
	Cla	ass I	Cla	ass II	Čla	ss III
	Thin	Hea∨y	Thin	Heavy	Thin	Heavy
A	2.0	1.5	1,5	1,0	1.5	1.0
В	2,5	1.5	1,5	1.0	1.5	1.0
С	2.0	1,5	1.5	1.0	1.5	1,0
D	2.5	1.5	2.0	1.5	2.0	1,5

### Specification RSC-AF-3a

This specification also applies to 9Ni-4Co steels, but to those possessing a nominal strength of 250,000 psi. The three classes were:

Class I, air melt, Si/Al-deoxidized steel

Class II, air melt, Si/Al-deoxidized, vacuum-arc-remelt steel

Class III, air melt, vacuum-arc-remelt steel, carbon-deoxidized.

Chemical Analysis (All Three Classes).

Element .	Percent		
C	0, 42-0, 48		
Mn	0.10-0.35		
Si	0.15-0.35 <sup>(a)</sup>		
P	0,01 max		
S	0,01 max		
Ni	7,0-8,5		
Co	3,5-4,5		
Mo	0.20-0.35		
Cr	0.20-0.35		
v	0.06-0.12		

(a) In carbon-deoxidized steel, 0.10% maximum silicon was specified.

### Heat Treatment.

Austenitize 1475 F, 1 hour, oil quench

Refrigerate at -100 F for 2 hours

Double temper 2 hours each at 400 F, air cool.

Mechanical Properties (All Three Classes).

### Mechanical Property

240 (min)
265 (min)
20 (min)
7 (min)

Magnetic-Particle Inspection.

	Class I	Class II	Class III
AMS Step-Down Test	2301-A	2300-A	2300-A
Frequency/Severity Rating (F/S)	(1.00/0.75)	0.50/1.00	0,50/1.00

Microcleanliness (J-K Rating to ASTM E45-61).

Inclusion Ratings (Worst Field)

Inclusion Type		#11011	ADAVII AVGGE		,	
	Cla	ass I	Cla	ass II	Cla	ss III
	Thin	Heavy	Thin	Heavy	Thin	Heavy
	2.0	3 5	1 5	1.0	1.5	1.0
- A	2.0	1.5	1.5			
В	2.5	1.5	1.5	1.0	1.5	1.0
С	2.0	1.5	1.5	1.0	1.5	1.0
D	2.5	1.5	2.0	1.5	2.0	1.5

### Other Qualifying Tests

Slices were cut from the top and bottom of all the billets used in the program. The slices were deep etched in hot acid and the subsequent macroexamination indicated that all were sound and of acceptable quality with respect to segregation, soundness, etc. All the billets were ultrasonically inspected.

### **ABBREVIATIONS**

Frequent repetition of a number of terms has made it convenient to use abbreviations in the text. These are listed below.

- (1) (200), (250), or (300) after steel designation Nominal yield strength in 1000 pounds per square inch
- (2) AM Air melt
- (3) VAR Vacuum-arc remelt
- (4) AM-D Air melt plus induction-stirred-ladle vacuum degassing
- (5) ISLVD Induction-stirred ladle-vacuum degassed
- (6) ESR Electroslag remelt
- (7) Si/Al-DOX Silicon-aluminum deoxidation
- (8) C-DOX Carbon deoxidation
- (9) HAZ Heat-affected zone
- (10) TIG Tungsten-arc inert-gas welding process
- (11) RCS Round-cornered square billet
- (12) PCC Precracked Charpy impact specimen
- (13) PTC Partial-thickness-crack (fracture-toughness testing).

### HEATS PRODUCED FOR THE PROGRAM

Table 1 summarizes the general processing procedures employed for the various heats produced for use in the program, and Table 2 lists the results of chemical analyses performed on samples taken from the billets.

TABLE 1. SUMMARY OF THE GENERAL PROCESSING PROCEDURES PRODUCED FOR THE VARIOUS HEATS EMPLOYED IN THE PROGRAM

Heat No.	Grade	Size, tons	Source	Melting Practices
08749	18Ni (300)	2	Vanadium-Alloys Steel Company	Air melted plus vacuum-arc remelted
3321284	18Ni (300)	70	Republic Steel Corporation	Air melted
X53201 X53088	18Ni (250)	85(a) 85(a)	United States Steel Corporation	Air melted
3321290	18Ni (250)	70	Republic Steel Corporation	Air melted plus induction-stitred ladie-vacuum degassed
3960882 3920864 3920872 3920873 3930871	18Ní <b>(250)</b>	13 4 4 4 2	Republic Steel Corroration	Air melted plus vacuum-arc remelted
C-425	18Ni <b>(</b> 250)	4	Firth Sterling	Air melted plus electroslag remelted
E-5565	9N1-4Co <b>(</b> 200)	15	Standard Steel	Air melted with silicon/auminum deoxidation
3920835	9N1 <b>-4</b> Co (200)	4	Republic Stee remelt	Air melted with silicon/aluminum deoxidation plus vacuum-arc remelting
3920795	9Ni-4Co (250)	4	Republic Steel remelt	Air melted plus carbon deoxidation during vacuum-arc remelting
3321246	9Ni <b>~4</b> Co <b>(</b> 250)	<b>7</b> 0	Republic Steel Corporation	Air melted with silicon/aluminum deoxidation plus induction-stirred-ladle vacuum degassing
3920851	9Ni-4Ça (250)	4 .	Republic Steel Corporation	Air melted with silicon/aluminum deoxidation plus vacuum-arc remelted
3920852	9Ni-4Co (250)	4	Republic Steel Corporation	Air melted plus carbon deoxidation during vacuum-arc remelting
3321262	9Ni-4Co <b>(</b> 250)	70	Republic Steel Corporation	Air melted plus carbon deoxidation during induction-sitrred-ladle vacuum degassing

<sup>(</sup>a) Only a small fraction of the heat was purchased for the program.

TABLE 2. CHEMICAL ANALYSES OF THE STEELS

Heat No.					Chemical Composition, percent	osition, per	rcent				
	U	Mn	Sí	a.	S	Z	ც	Ψ°	V.	Ţ	Othe:
				18Ni	18Ni (300) VAR						
RSC-AF-1 Class III(2)	0.03 (max)	0	0	ò	9	ñ	00	4	0.05/0.15	0.60/0.90	<b>3</b>
08749 (2 tons)	0.025	0.03	0.05	0.004	0.004	19.05	9.30	4.55	0, 16	0.58	
3321284 (AM 70 tons)	0,024	0.13	0,10	0,003	0.005	18,50	8.80 80	4,93	0.13	0.13	
				18Ni-Co	18Ni-Co-Mo (250) AM						
RSC-AF-1 Class I	0.03 (max)	0.10 (max)	0.10 (max)	0 0.010 (max)	x) 0.010(max)	17,5/18.5	5 7.5/8.5	4.7/5.2	0.05/0.15	0.30/0.50	2
X53201 (85 tons)	0.020						<u>5</u>	4.85	0.16	0.49	
X53088 (85 tons)	0,022	90.0	0.03	0.006	0.010	17.10	8,20	4.95	<b>5</b> .0	0.39	
3321290 (ISLVD) (70 tons)	0.025	0,10	0.07	0.005	0° 00 <del>4</del>	17.93	7.85	5, 03	0, 13	0.49	
				18N	18N1 (250) VAR						
RSC-AF-1 Class II	0.03 (max)	0.10 (max)	0, 19 (max)	0 0.010(max)	x) 0.010(max)	17.5/19.5	5 1.5/8.5	4. 75.2	0.05/0.15	0.30/0.50	3
3920864 (4 tons)	0.03			0.004	0,004		7.85	5,03	0, 12	0,50	
3930871 (2 tons)						;	,	,			
3920872 (4 tons)	0.019	0.08	0.04	900.0	0,003	17,88	7,78	4, 95	0, 11	0.49	
3920873 (4 tons)	0.019	ල් ජ	90.0	g, 004	0,005	17.86	7.78	4, 99	0, 11	0.50	
3960882 (13 tons)	0.022	0.08	0,08	0.004	0.003	17.85	7.75	4.95	0.11	0,47	
C-425 (ESR) (4 tons)	0.019	0.11	0.02	0.005	0,005	17.78	7.81	4.98	0.07	0.47	
	U	Mn	Šį	ć.	တ	ž	ဒ	Мо	ö	ا ح	>
				9Ni -₄C	9Ni -4Co (200) AM						
RSC-AF-2A. Class I	0.24/0.30	0,10/0.35	0.20/0.35	0,010 (max)	0.010 (max)	7.0/8.0	3.5/4.5	0,35/0,60	0.35/0.6	(S)	0.06/0.12
E-5565 (15 tons)	0.27	0.46	0.30	0,006	0,004	7.65	3.80	0.45	0,56	0,03	0,09
				9Ni -4C	9N1-4Co (200) VA3						
RSC+AF-2A Class II 3920835 (4 tons)	0.24/0.30 0.28	0,10/0,35 0,24	0.20/0.35 ( 0.28	0.010 (max) 0.005	0.010 (max) 0.005	7.0/8.0	3.5/4.5 ( 3.81	0.35/0.60	0.35/3.60 0.56	(c) 0.00 0.02	0,06/0,12 0,09
				9Ní -4C	9Ni-4Co (200) VAR						
NC-AF-2A Class III	0.25/0.30	0	0.10 (max) 0.010 (max)	), 010 (max)	0.010 (max)	0	2	0,35/0,60	0,35/0,60		0.06/0.12
3920795 (4 tons)	0.246	0.22	0,01	0,004	90.00	8, 05	3.90	0,55	0.52	0.01	8

TABLE 2. (Continued)

Heat No.				J	Chemical Composition, percent	osttion, per	cent				
	U	Mn	Si	6	s	ž	ပိ	Мо	Ö	₹	>
				N N	9NI-4Co (250) AM						
RSC-AF-3A Class I 3321246 (70 tons)	0.42/0.48 0.43		0.10/0.35 0.15/0.35 0.15 0.28	0,010 (max) 0,003	0.010 (max) 0.010 (max) 0.003 0.007	7.0/8.5	3.5/4.5	0.20/0.35	0,20/0,35 (c) 0,27 0,03	0°03	0.06/0.12 0.09
				9-1NG	9Ni-4Co (250) VAR						
RSC-AF-3A Class II 3920851 (4 tons)	0.42/0.48		0, 15/0, 35 0, 28	0.010 (max) 0.004	0.10/0.35 0.15/0.35 0.010 (max) 0.010 (max) 0.14 0.28 0.004 0.007	7.0/8.5	3.5/4.5	0.20/0.35	0,20/0,35 0,28	o.03	0, 06/0, 12 0, 09
				- Ne	9NI-4Co (250) VAR						
RSC-AF-3A Class III 3920852 (4 tons)	0.42/0.48 0.44	0,10/0,35 0,13		0.010 (max) 0.005	0.10(max) 0.010(max) 0.010(max) 0.01 0.005 0.008	7.6/8.5	3.5/4.5 3.98	0.20/0.35 0.31	0.20/0.35	0.01	0, 06/0, 12 0, 09
				-ive	9Ni-4Co (250) AM						
RSC-AF-3A Class III 3321262 (70 tons)	0.42/0.48 0.45		0.10 (max) 0.02	0.010 (max) 0.004	0.10/9.35 0.10(max) 0.010 (max) 0.010 (max) 0.17 0.02 0.004 0.008		3.5/4.5 3.93	7.0/8.5 3.5/4.5 0.20/0.35 7.80 3.93 0.30	0.20/0.35	0.01	0.06/0.12
(a) Specifications RSC-AF-1, 2A and 3A. (b) Added 0.003% B. 0.02% Zr, 0.05% Ca. (c) Aluminum added for grain refiner ent and deoxidation.	0.02% Zr. 0. for grain refin	nd 3A. 05% Ca. nerrent and d	eoxí dation.								

### Sources of the Materials

Most of the steel used in the program was produced by Republic Steel Corporation. Some additional material was purchased from other steel producers to permit a comparison to be made of the properties of similar products made at various steel mills and to determine whether the results of the evaluations performed were limited to the product of a single producer. The other steel companies that produced material for the program, together with a description of the processing used, are indicated in the following paragraphs.

United States Steel Corporation, Duquesne Works, produced an air-melted 36 x 36-inch bloom ingot of 18Ni (250) steel from the 85-ton Heat No. 73201. It was forged to 11 inches square and machine conditioned to 10 inches square before shipment to Republic Steel Corporation. The steel conformed to Specification RSC-AF-1 Class I. Part of an additional 85-ton heat, Heat No. 53088, was also supplied by U. S. Steel Corporation to Republic Steel Corporation as 3/4-inch plate and further processed to 3/8-inch plate and 0.045 and 0.160-inch plate by Republic.

Vanadium-Alloys Steel Company, Latrobe, Pennsylvania, produced Heat No. 08749 as a 20-inch-diameter, 4,275-pound vacuum-arc-remelted ingot of 18Ni (300) steel conforming to Specification RSC-AF-1 Class III. Part of the ingot was shipped to Republic as an 8 by 16-inch billet, and part as 0.045-inch and 0.160-inch-thick sheet.

Lebanon Steel Foundry, Lebanon, Pennsylvania, produced Heat No. W-1443 of 9Ni-4Co (200) steel. It was made in a 5-ton electric furnace to melting specifications supplied by Republic. No silicon or aluminum was used to deoxidize the steel, and the entire heat was poured as "open" steel into a 21-inch-diameter by 9,500-pound electrode-ingot mold. The electrode ingot was then vacuum-arc remelted by Republic Steel to produce the 24-inch-diameter by 8,500-pound ingot, Heat No. 3920795. During the vacuum-arc-remelt process, the steel was deoxidized by the carbon-oxygen reaction. The heat conformed to Specification RSC-AF-2A Class III.

Standard Steel Division of Baldwin-Lima-Hamilton Corporation at Burnham, Pennsylvania, produced Heat No. E-5565 of 9Ni-4Co (200) steel in a 15-ton electric furnace to melting specifications and practices supplied by Republic Steel. The heat was melted in air and deoxidized with silicon and aluminum additions in the furnace and ladle. It conformed to Specification RSC-AF-2A Class I. One 25 x 27-inch by 12,000-pound and one 21-inch-diameter by 9,500-pound electrode ingot were cast. The 25 x 27-inch ingot was processed and tested as an air-melt heat. The electrode ingot was vacuum-arc remelted by Republic Steel to produce the 24-inch-diameter by 8,500-pound ingot (Heat 3920835), which conformed to Specification RSC-AF-2A Class II.

Firth Sterling, Incorporated, Pittsburgh, Pennsylvania, provided Heat C-425 (ESR) of 18Ni (250) steel conforming to Specification RSC-AF-1 Class II. It was prepared from two 14-inch-diameter x 4,000-pound electrodes forged from an air-melt  $25 \times 27$ -inch ingot from Heat 3321290 produced by Republic Steel Corporation. The electrodes were electroslag arc remelted (ESR) by Firth Sterling to produce one 20-inch-diameter x 8,000-pound (nominal weight) ingot, which was identified as Heat C-425 (ESR). The ESR ingot was forged by Republic Steel to a  $16 \times 8$ -inch billet for subsequent testing.

### Melting Procedures

An important objective of the work was to determine the most desirable melting and processing procedures for the production of the 18Ni maraging and 9Ni-4Co quench and temper steels. To provide material for evaluating the influence of various melting practices and processing techniques upon the mechanical properties and reliability of the steels, heats were prepared by the practices listed below. They were:

- (1) Air melted in basic electric furnace (AM)
- (2) Air melted and induction-stirred-ladle vacuum degassed (AM-D)
- (3) Remelted in vacuum consumable-electrode arc furnace (VAR)
- (4) Electroslag remelted in consumable electric-arc furnace (ESR).

Practices were varied to include silicon-aluminum deoxidation of both air-melted and vacuum-arc-remelted steels and carbon deoxidation during vacuum degassing and vacuum-arc remelting. The steels were cast into ingot molds of various sizes to provide material for subsequent processing and also to determine the influence of ingot size upon the metallurgical structures and mechanical properties of thick plate.

### Seventy-Ton Electric-Arc-Furnace Practice

Both the 18Ni maraging steel and the 9Ni-4Co steel were melted by Republic Steel in a 70-ton electric furnace. Scrap charges for the first 70-ton heat of the maraging and the quench and temper steels were normal for aircraft-quality alloy material melted to low phosphorus and sulfur contents. Scrap charges for subsequent 70-ton heats of the two grades of steels were made up of surplus steel from preceding heats produced for the program, plus the additional low-phosphorus light scrap needed to make up the charge.

The 70-ton heats were milted down under a normal lime-spar oxidizing slag. When the charge was completely melted, iron ore or roll scale was added to promote early active oxidation. After periodic flushing and remaking of the slags, the 'eats were blown with dry, gaseous oxygen to reduce the carbon contents to the desired levels of 0.02 percent or less for the 18Ni steels and approximately 0.10 percent below specification for the 9Ni-4Co steels. The lime-spar slags were then removed and finishing slags made up to aid in desulfurization and deoxidation.

The 18Ni maraging steels were finished under a lime-alumina slag. During the finishing period, sulfur was reduced to about 0.006 percent or less before final chemistry adjustments were made. The slag was then reshaped and the bath temperature adjusted. At this point, the 18Ni Heat No. 3321290 was transferred to the vacuum-degassing ladle, then deoxidized with Ca, Zr, and B before pouring. The other 70-ton 18Ni Heat No. 3321284 was partially deoxidized in the furnace by additions of Zr, Ca, and B, preparatory to subsequent remelting in the vacuum-arc furnace.

The 9Ni-4Co (250)-grade steels were finished under a carbide slag. This applied to both conventional silicon-aluminum-deoxidation and to carbon-deoxidation practices. After the slag was formed, the alloy composition was adjusted to melting specifications. For the silicon-aluminum heat (Heat No. 3321246), the silicon was added in the furnace

and the bath temperature adjusted for tapping. The heat was then transferred to the induction-stirred vacuum-degassing ladle. Aluminum was added to the ladle just prior to teeming. For the carbon-deoxidized heat (3321262) the carbide-type slag was made only mildly reducing. No deoxidizers were added in the furnace, with the result that the melt had a relatively high oxygen content at the time it was introduced into the degassing ladle.

All four of the 70-ton heats made at Republic were teemed under an argon shield. Details on the ingot sizes produced from four 70-ton heats are given below.

Heat No. 3321290 - 18Ni (250) (ISLVD) steel. The scrap charge for this heat was of 18Ni (300) steel grade. The heat was air melted, then poured utilizing Republic's induction-stirred-ladle vacuum-degassing procedure. Steel was cast into two 43-inch x 23,000-pound octagon ingots, four 25 x 27-inch x 12,000-pound ingots, one 28-inch-diameter x 25,000-pound, and three 21-inch-diameter x 9,000-pound electrode ingots.

Heat No. 3321284 - 18Ni (300)(AM) steel. The heat was air melted from a scrap charge of 18Ni (300) steel. It was cast into three 21-inch-diameter x 9,000-pound electrode ingots.

Heat No. 3321246 - 9Ni-4Co (250)(Si/Al-DOX) steel. The scrap charge was normal for aircraft-quality alloy steel. The heat was silicon "blocked" in the furnace, then vacuum degassed. A small aluminum addition was made to the ladle just prior to teeming. Two 24 x 27-inch x 12,000-pound ingots and one 21-inch-diameter x 9,000-pound electrode ingot were cast.

Heat No. 3321262 - 9Ni-4Co (250)(C-DOX) steel. The scrap charge was 9Ni-4Co (250) steel. The heat was poured in the "open" condition and deoxidized by the carbon reaction in the ladle degasser. Two  $24 \times 27$ -inch  $\times 12$ , 000-pound ingots and one 21-inch-diameter  $\times 9$ , 000-pound electrode ingot were cast.

### Vacuum-Arc Remelting

A number of electrode ingots were remelted in a consumable-electrode vacuumarc furnace. The effect on gas content (H2, N2, and O2) is indicated below.

		Gas	Content,	ppm
Heat No.	Steel Grade and Melting Practice	H <sub>2</sub>	N <sub>2</sub>	02
E-5565	9Ni-4Co (200) AM-Si/Al-DOX	0.2	32	70
3920835	9Ni-4Co (200) VAR (Parent Heat No. E-5565)	0,1	4	12
3321246	9Ni-4Co (250) AM-D-Si/Al-DOX	0.2	24	20
3920851	9Ni-4Co (250) VAR (Parent Heat No. 3321246)	0.2	16	4
3321290	18Ni (250) AM-D	1.3	16	8
3920865	18Ni (250) VAR (Parent Heat No. 3321290)	0.9	11	9

It is evident that the oxygen and nitrogen contents were lowered substantially during the vacuum-arc remelting of the two 9Ni-4Co steels. Vacuum-arc remelting had little effect on the 18Ni steel, presumably because of its low carbon content.

The following consumable-electrode vacuum-arc-remelted heats were prepared by Republic:

			Ingot Size		
Heat No.	Grade	Parent Heat No.	Diameter, in.	Weight, lb	
3920852	9Ni-4Co (250)	3321262	24	8,500	
3920851	9Ni-4Co (250)	3321246	24	8,000	
3920795	9Ni-4Co (200)	W-1443	24	8,500	
3920835	9Ni-4Co (200)	E-5565	24	8,500	
3920864	18Ni (250)	3321290	24	8,500	
3960882	18Ni (250)	3321290	32	25,000	
3920872	18Ni (250)	3321290	24	8,500	
3930871	18Ni (250)	3321290	18	4,500	
3920873	18Ni (250)	3321290	24	8,500	

### Electroslag Remelting Practice

One ingot, Heat No. C-425, was prepared by Firth Sterling, Incorporated, using the electroslag process. In the preparation of this ingot, two 14-inch-diameter ingots from Heat No. 3321290 were remelted under a conductive slag in a consumable-electrode arc furnace. The process afforded the advantage of limited alloy adjustment but did not provide for degassing since melting was done without vacuum.

Post-Teeming Treatment. The 18Ni maraging steel ingots were not given a post-teeming treatment. Experience at Republic Steel with electrode ingots of this grade of steel showed them not to be subject to cracking when air cooled after stripping from the ingot molds. This freedom from cracking was attributed to the softness of the martensite formed on transformation during cooling. Ingots were stripped from the molds 3 to 8 hours after teeming and then permitted to air cool to room temperature.

On the other hand, the 9Ni-4Co (250) ingots required post-teeming treatment to minimize the probability of cracking from thermal stresses and structural changes. Ingots of the steel were stripped from the molds 3 hours after teeming and cycle cooled by (1) soaking at 1250 F for 20 hours, (2) furnace cooling to 1000 F, and (3) air cooling to room temperature.

The electrode ingots cast from air-melted 9Ni-4Co steel, however, were not given a post-teeming treatment since crack defects were not considered detrimental to remelting. Such ingots were held in the mold for a minimum of 2 hours, then stripped and conditioned for vacuum-arc remelting.

The 9Ni-4Co (200) air-melt ingot (Heat No. E-5565) produced by Standard Steel was reported to have been cycle cooled by soaking at 1550 F for 5 hours, cooling at 50 F/hr to 1100 F, soaking at 1100 F for 5 hours, cooling at 50 F/hr to 200 F, and air cooling to room temperature.

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# QUALIFYING-TEST RESULTS

Ingots from ten on heats listed in Table 1 were forged to 8 x 16-inch billets, and material cut from these billets was used for the qualifying tests described previously. In addition, specimens cut from the 10-inch-square billet of Heat No. X53201 supplied by U.S. Steel were subjected to the qualifying tests. The 11 billets were subjected to ultrasonic inspection. The billet from Heat No. E5565 was found to be cracked. However, it was possible to crop the billet to sound metal and proceed with the testing. Transverse slices cut from the billets were deep etched, and all 11 were found to be satisfactory. Other slices cut from the billets were reforged to 5-inch squares, and the 5-inch squares were then cut up for tensile tests, chemical analysis, gas analysis, microcleanliness, magnetic-particle inspection, Jominy hardenability testing, and grain-size determination. Results of the chemical analysis were presented in the previous section as Table 2.

Tensile-test results are summarized in Table 3. It may be noted from Table 3 that the tests on Heats No. 08749, No. 3321246, and No. 3920851 did not meet the specified ductility under all conditions. Heats No. C425 and No. X53201 failed by a small amount to meet the specified strength. Since there was no assurance that remaking the heats would result in better properties, the heats were accepted in spite of the deviations.

Magnetic-particle and microcleanliness tests for the 11 billets fell within the specification with one exception. Heat No. E5565, upon magnetic particle inspection, had a frequency-severity rating slightly above the specification. Its rating for Type B and D inclusions in the microcleanliness test was also slightly outside the limits of the specification. Neither of these deficiencies was considered sufficiently important to justify remaking the he however.

# INGOT FORGING

Except for the steel supplied by U.S. Steel Corporation and Vanadium-Alloy Steel Company, ingoi breakdown was done on Republic's 3000-ten Birdsboro forging press located in Canton, Ohio.

#### 18Ni-Steel

Based on the initial literature survey and prior experience with maraging steels, the following processing was specified for all three classes of this material:

- (1) Freheat to 1200 F
- (2) : eat to 2250 F and soak for sufficient time to achieve temperature uniformity
- (3) Forge until cooled to 1700 to 1900 F
- (4) Reneat as needed to 2200 to 2250 F

TABLE 3. TENSILE-PROPERTIES HEAT-QUALIFICATION TESTS

Heat No.	$\frac{Test}{Direction^{\{a\}}}$	Yield Strength, ksi	Ultimate Tensile Strength, ksi	seduction of Area, percent	Elongation, percent	Hardness, RC
		STAGI	PM: Stool		İ	
		Not	1000			
	E	272.0	280. 2	16.7	3, 0	50.0
08749 VAR (Class LL)	1	• • • • • • • • • • • • • • • • • • •	0 370	44 8	0 6	46.0
X53201 AM (Class I)	J	238.U	0.047	) (		. 07
	F	250.6	264, 7	32, 0	0.7	48.5
332190 ISLVD (CIASS 1)	<b>-</b> [	250.5	2 % R 1	32. 7	6, 0	46.0
3920854 VAR (Class II)	<b>—</b>	70.00	1.003	. (	•	
C-425 ESR (Class II)	H,	239.6	252. 7	37.0	o. s	4%.0
	•	9-iN9	9Ni-4Co Steel			
	•	0 101	209. 5	55.0	14.0	42.5
E5565 AM (Class 1)	۱ د	0.101	0 202	57.0	15.0	38.0
3920835 VAR (Clas.: II)	<b>,</b>	10%	» «	0 67	0 41	38.0
3920795 VAR (Class III)	H	181.2	193.0	070	0.01	
2221246 446 (Class !!)	Ę	250.6	297.8	18.0	9.0	51.0
2271740 AM (Class II)	4 1	, , ,	303 3	23.0	6.0	51.0
392085i VAR (Class II)	E⊣	9.757	306. 2	3	· c	0 0 0
3920852 VAR (Class III)	Ή	251.6	296.8	30.0	0.0	47.0

(a) i indicates transverse test, top of billet; L indicates longitudinal test.

- (5) Finish forging at 1700 to 1900 F
- (6) Air cool.

The above schedule was used without difficulty in forging several 8 x 16-inch billets by Republic, by U. S. Steel Corporation in producing the 10-inch-square billet of Heat No. X53201 and by Vanadium-Alloy Steel Company in producing the 8 x 16-inch billet from Heat No. 08749. However, cracking was encountered in producing two 30-inch, round-cornered-square (RCS) blooms forged by Republic from two 43-inch ingots of Heat No. 3321290. Previous to this, a 25 by 27-inch ingot from this same heat had been forged to an 8 x 16-inch billet without difficulty. The development of cracks in the 30-inch RCS blooms caused Republic to undertake a detailed examination of the cracked blooms. It was concluded that the cracking was the result of a combination of transformation and thermal stress plus a small degree of hydrogen embrittlement. On the basis of this examination, the following revisions were made in heating and cooling associated with ingot forging:

- (1) Heat to 2300 F (rather than 2250 F) and soak for a minimum of 3 hours
- (2) Use ingots large enough to permit at least a 3 to 1 reduction in cross section
- (3) Air cool to 1200 F, then bury in vermiculite until the billet or bloom reaches a temperature of 200 F
- (4) Heat to 300 to 500 F for approximately 36 hours before grinding or stocking.

After these changes in ingot forging were placed into effect, no further difficulty was encountered in forging the 18Ni steels.

#### 9Ni-4Co Steel

The initial practice specified for forging (including ingot breakdown) of all three classes of 9Ni-4Co steel was based on Republic's previous experience with vacuum-arc-remelted steel and the literature survey conducted as the first phase of the program. It called for:

- (1) Preheating to 1200 F
- (2) Heating to 2050 F and soaking
- (3) Forging to size, reheating as required
- (4) Burying in vermiculite until cooled to at least 300 F
- (5) Air cooling.

It was found that cracks were likely to develop during the initial breakdown when this practice was followed. The practice was changed to the following:

- (1) Surface conditioning the ingot by grinding
- (2) Preheating to 1200 F

- (3) Heating to 2200 F and soaking
- (4) Forging to size, reheating to 2050 F as required
- (5) Burying in vermiculite until cooled to at least 300 F
- (6) Air cooling.

After making these revisions in the practice, no further cracking was experienced.

# Flame-Cutting Experiments on 18Ni Billets

During processing of the 18Ni-maraging steels for qualification purposes, macroctch sections through flame cut surfaces showed extensive cracking in both the 8-inch x 16-inch billets and the 5-inch-square reforged test samples taken from the top and bottom of the air-melted Heat No. 3321290. Later, similar cracks were observed in specimens from air-melted Heat No. 53201. These observations led to a series of tests to establish procedures that would eliminate cracking. Three approaches were followed:

- (1) Heating to 250 F, 450 F, and 650 F and flame-cutting while the 5-inch test specimens were at these temperatures
- (2) Heating to 1100 F and cooling to room temperature prior to flame cutting
- (3) Flame cutting as the sample cooled from 1500 F.

The results indicated that preheating to 650 F or less was ineffective in preventing cracking. No cracking was observed when the specimens were heated to 1100 F, then cooled to room temperature before cutting, nor when specimens were cut while cooling from 1500 F. Flame cutting while the steel is several hundred degrees above room temperature introduces difficulties, and for this reason pretreatment at 1100 F was considered the more practical solution. In later work, the 18Ni steel was flame cut only after having been given a pretreatment at 1100 F. Most specimens were saw cut.

#### FORGEABILITY EVALUATIONS

Two separate but related investigations on forgeability were conducted. One was concerned with the effect of finishing temperature and the other was concerned with the effect of degree of red-ction.

#### Forging Finishing-Temperature Study

The forging temperature studies were performed at Republic Steel Corporation, on machined 4-inch-square x 5-inch blanks cut from 8 x 16-inch section billets of the 18Ni and 9Ni-4Co alloy steels. They were upset forged in a 500-ton press to

1-inch-thick pancakes in a single operation within 1 minute after the steel was removed from the furnaces. Upsetting was done at 50 F increments from 1750 F to 2000 F. These temperatures were selected as below those for ingot forging and characteristic of finishing temperatures for production forging. The forged pancakes of the 18Ni steels were air cooled from the forging temperatures; those from the 9Ni-4Co steels were slowly cooled by burying them in vermiculite.

Evaluation of the upset pancakes included the following tests:

- (1) Visual examination of pancakes
- (2) Macroetch of upset sections
- (3) Microscopic examination
- (4) Tensile tests (radial direction)
- (5) Fracture toughness tests (precracked Charpy-radial direction).

# Macroscopic and Microscopic Examinations

Visual examination of the as-forged pancakes revealed no abnormal surface discontinuities, such as surface and edge ruptures, for any of the steels. Similarly, the deep-etched macro specimens cut from the as-forged pancakes did not exhibit any significant differences except in the case of those from the 9Ni-4Co AM steel from Heat No. 5565. Some of these pancakes contained cracks, but the cracking did not correlate with forging temperature. A tendency to crack had been apparent in all forging operations of Heat No. E5565, beginning with the initial ingot breakdown. However, no difficulties were encountered in forging vacuum-arc-remelted steel (Heat No. 3920835), from parent Heat No. E5565, or with the 0.42 percent carbon, 9Ni-4Co(250) AM-Si/Al-Dox steel (Heat No. 3921246). It was therefore suggested that the tendency to cracking of Heat E5565 was a characteristic of that specific heat and not a characteristic of the steel grade.

An excellent correlation of as-forged grain size with the pancake forging temperature was observed for the 18Ni steels. A fibrous microstructure with greatly elongated grains was produced at 1750 F and 1800 F. At 1850 F, the structure was a mixture of equiaxed and elongated grains. At 1900 F and higher, the grain structure was completely equiaxed in both the radial and tangential directions. Rapid grain growth between 1950 F and 2000 F was indicated by the large grain size of pancakes forged at 2000 F.

A correlation of microstructure with finish forging temperatures of the 9Ni-4Co steels was not so marked as it was for the 18Ni steels. The fibrous elongated grain structure so evident in some 18Ni disks was not found in any of the 9Ni-4Co pancakes, even those forged at 1750 F. A gradual structure coarsening occurred in pancakes forged between 1750 F and 1950 F, while rapid structure coarsening occurred between 1950 F and 2000 F. The tests suggested that there might be some advantage in not permitting the finishing temperature to exceed 1950 F.

#### Tensile-Test Properties

The tensile properties were determined from specimens oriented radially in the pancakes after being heat treated as required by Specifications RSC-AF-1, 2A and 3A. A significant improvement in the ductilities of the materials tested was observed as compared with ductility values obtained earlier on the materials during the qualification tests.

The tensile-test result for the 18Ni steels revealed no significant variations of strengths and ductilities as related to the temperature at which the pancake was upset. Microscopic examination of test specimens showed that the as-forged microstructures of the steels were not radically altered by the heat treatment specified. The tensile-test results for the 9Ni-4Co steels likewise did not reveal significant variations of strengths and ductilities as related to forging finishing temperatures.

#### Fracture-Toughness Tests

Precracked Charpy impact tests were conducted on specimens removed from the forged pancakes with the thought that they might provide a more sensitive indication of the influence of forging finishing temperatures upon the properties of the 18Ni and 9Ni-4Co steels. Duplicate test specimens were tested. Some of the specimens were cut with the fatigue crack lying in a radial plane of the forged coupon and some with the fatigue crack in a tangential plane. In a few cases, radial and tangential tests were cut from the same coupon. The results were reported as W/A data (energy absorbed divided by the uncracked area).

The only differences in precracked-Charpy values for forged coupons was found to have a high degree of probability of being statistically significant were those for the three heats of 9Ni-4Co(200) steels. The data are summarized below:

Heat No.	Processing	$W/A^{(a)}$ , inlb/in. <sup>2</sup>
E5565	Am-Si/Al-DOX	1320
3920835	Si/Al-DOX-VAR	2050
3920795	VAR-C-DOX	376ú

(a) Each value is the average of 12 tests. Two specimens were cut from each of a series of 6 forging coupons made on each heat.

Heat No. 3920795 was first melted by Lebanon Steel Foundry as a 5-ton electric furnace Heat No. W1443 and cast as "open" steel (no deoxidation of any kind) into a 9,500-lb ingot. The ingot was remelted by Republic in a vacuum-arc furnace, with only carbon deoxidation. The final ingot contained less than 0.01 percent. Heat No. 3920835 was made by remelting one ingot of air-melted Heat No. 5565 (Standard Steel) in Republic's vacuum-arc furnace. Heat No. 5565 contained 0.30 percent Si and 0.03 percent Al, and the remelted ingot (Heat 3920835) contained 0.28 percent Si and 0.02 percent Al. The results suggest that fracture toughness is improved by vacuum-arc remelting and that silicon and aluminum additions should be avoided. The three heats of 9Ni-4Co(250) (0.45 percent carbon) exhibited a similar but less proneunced trend.

#### Finishing Temperature

The orging-temperature tests on the coupons described in the foregoing paragraphs led to the selection of the following finishing temperatures for later forging-reduction studies and for later sheet and plate studies:

- (1) 18Ni steel, either air melted or vacuum degassed, was to be finished at 1900 to 1950 F
- (2) 18Ni steel, vacous a citeralized. FF to be finished at 1850 to 1900 F
- (3) 9Ni-4Co steel, of all types, was to be first dat 1900 to 1950 F.

# Forging-Reduction studies

The forging-reduction studies, conducted by General Dy. mics/Fort W orth under subcontract to Republi. Steel Corporation, were exsigned to do ermine the minimum amount of hot working required at the finishing temperature as well as the best menting practice to provide maximum reproducible mechanical and fracture-toughness properties in production-forged parts. Three percentages of hot working = 25, 50, and 75 percent - were investigated. Results of the study were evaluated on the basis of room-temperature tensile properties, fracture toughness at room temperature and -65 F, impact properties at -65 F, and metallographic studies.

#### Materials

The four steel alloys were received at General Dynamics in t'e form of 3 sizes of forged billets. A total of 27 billets, representing 9 heats of steel, were evaluated. The billets were flat plate forgings nominally 16 inches wide in random lengths, and were 2, 4, and 6 inches thick. The three thicknesses represented hot-forging reductions from the original 8 x 16-inch cross sections of 75, 50, and 25 percent, respectively. The 9 heats a sed in this part of the program, along with the grade designation and ingot size, were as fallows:

Code	Heat No.	Grade	Ingot Size
A	3920795	9Ni-4Co(200) VAR-C-DOX	24-inch diameter
В	E5565	9Ni-4Co(200) AM-Si/Al-DOX	25 x 27 inches
С	3920835	9Ni-4Co(200) VAR	24 inch diameter
$\mathbf{a}$	3920852	9Ni-4Co(250) /AR-C-DOX	24-inch diameter
E	3321246	9Ni-4Co(250) AM-Si/Al-DOX	$25 \times 27$ inches
r	3920851	9Ni-4Co(250) VAR	24-inch diameter
G	3321290	18Ni (250) AM-D	25 x 27 inches
H	3920864	18Ni (250) VAR	24-inch diameter
J	08749	18Ni (300) VAR	20-inch diameter

All of the specimens of each grade of steel were heat treated prior to evaluation as prescribed by Republic Steel Corporation on the basis of previous studies in this

program. The 18Ni maraging steel was solution annealed at 1500 F for 1 hour per inch of minimum thickness. After the test specimens were finish machined, they were aged at 925 F for 3 hours.

The 9Ni-4Co(250) steel was evaluated in two different heat-treated conditions, a martensitic and bainitic condition. The martensitic condition was designed to produce a very high yield strength, approximately 250 ksi. The bainitic heat treatment was intended to improve toughness while maintaining an ultimate strength of about 260 ksi.

The martensitic heat treatment of the 9Ni-4Co(250) steel was accomplished as follows:

- (1) Austenitize 1 hour at 1475 F, quench in warm oil (140 F)
- (2) Refrigerate 2 hours at -100 F in Dry Ice-acetone mixture
- (3) Temper 2 hours at 475 F
- (4) Final machine or grind
- (5) Temper 2 hours at 475 F.

The bainitic heat treatment was accomplished as follows for the 9Ni-4Co(250) steel:

- (1) Austenitize I hour at 1475 F
- (2) Quench into a molten-salt bath at 485 ± 10F and hold for 6 hours
- (3) Air cool no stress relief after final machining or grinding of specimens.

The following heat treatment was used for the 9Ni-4Co(200) steel to achieve the desired 200-ksi ultimate strength;

- (1) Normalize I hour at 1650 F, air cool
- (2) Austenitize I hour at 1550 F; quench in warm oil
- (3) Double temper for 2 hours each at 1000 F.

Typical hardnesses produced after these heat treatments are given in Table 4 for the steels produced by different melting practices. For each grade and condition, the steels produced by vacuum-arc remelting (VAR) appear slightly softer. This may result from lower interstitial contents in these steels.

#### Tensile Properties

Tensile tests were conducted on 0. 12-inch-diameter solutions by procedures satisfying requirements of Federal Test histhod 151a, Method 211.1, and ASIM E8-61T. The tests were made on a 120,000-lb-capacity Raldwin Universal testing machine equipped with a Model MA-1 load-deformation recorder. The Standard 0. 252-inch-diameter specimens were load paced at 18,000 lb/min (approximately 90,000 psi/min) to the yield point. Yield points were calculated by the 0.2 percent offset method.

TABLE 4. TYPICAL ROCKWELL C HARDNESSES OF THE 9Ni-4Co AND 18Ni-Co-Mo STEELS IN THE HEAT-TREATED CONLITION

Ailoy	Melting Practice	Code	Hardness, Rockwell C <sup>(a)</sup>
9Ni-4Co(200)	VAR-C-DOX	A	42.4
	AM-Si/Al-DOX	В	44.0
	AM-Si/Al-DOX-VAR	C	44.3
9Ni-4Co(250)	VAR-C-DOX	D	51.7
Bainite	AM-Si/Al-DOX-ISLVD	E	51.9
	Si/Al-DOX-VAR	E.	51.7
9Ni-4Co(250)	VAR-C-DOX	D	53. 1
Martensite	Si/Al-DOX-ISLVD	E	54, 1
	Si/Al-DOX-VAR	F	54. 2
18Ni(250)	AM-ISLVD	G	52.0
	VAR	H	51.7
18Ni(300)	VAR	J	53.5

<sup>(</sup>a) Average of five tests; hear treatments are described in the text.

Data on the average mechanical properties of the 9Ni-4Co and 18Ni-Co-Mo steels are summarized in Table 5 for tests conducted at room temperature. These data cover only the properties obtained for the optimum forging reduction for each steel. Table 6 compares the steels by grade relative to the air-melt practice. The properties of the air-melted steels of each grade are rated 1.00 and the properties of steels produced by other melting practices are compared to the properties of the air-melted steels. The values compared are the properties obtained with the optimum forging reduction for the melt practice.

For the 9Ni-4Co(250) steels in the bainitic condition, the yield strengths are lower than anticipated. Only the VAR-C-DOX steel attained a yield strength of 220 ksi; the steels produced by other melting practices had lower yield strengths. Tensile strengths for these steels are higher than 260 ksi, as can be seen in Tables 3 and 5.

In the martensitic condition, yield strengths of the 9Ni-4Co(250) steels ranged from 243 to 256 ksi and tensile strengths from 280 to 297 ksi. The tensile and yield strengths of the VAR-C-DOX steel (D), reduced 75 percent in finish forging, were about 11 and 15 ksi lower, respectively, than either the Si/Al DOX-ISLVD(E) or Si/Al-DOX-VAR(F) steels.

The yield-strength and ductility values of the 9Ni-4Co(200) steel produced by the VAR-C-DOX rielting practice (A) and reduced 25 percent in finish forging were higher than those for steels produced either by AM-Si/Al-DOX (B) with 25 percent reduction or Si/Al-DOX-VAR (C) with 75 percent reduction. Tensile strength of the VAR-C-DOX steel (A), however, is lower than that of the other two steels.

A combination of VAR steel (H) and 75 percent reduction in finish forging produced significantly higher yield and tensile strengths in the 18Ni(250) steel than was obtained by AM-ISLVD (G) melting practice. Ductilities of the two steels were very nearly equal.

TABLE 5. AVERAGE MECHANICAL PROPERTIES OF 9NE-4Co AND

				Best					Longitudinal
Mate	rial	Code	Melt Practice	Forging Reduction, percent	Fty,	Ftu, ksi	Elongation, percent in 2 in.	Reduction in Ares, percent	Impact <sup>(a)</sup> , ft-1b
9Ni-4Co(2	50)								***************************************
Bainite	•								
Heat No.	3920852	D	VAR-C-DOX	75	220.1	261.4	12.0	53.2	22.9
	3321246	E	Si/Al-DOX- ISLVD	50	213.3	263.6	12.0	41.9	15.6
	3920851	F	Si/Al-DOX- VAR	75	209.1	262.5	12.7	41,8	22.0
Martensia	e								
Heat No.	3920852	D	VAR-C-DOX	75	243.0	280.1	11.0	44.0	14.0
	3321246	Ε.	Si/A1-DOX- ISLVD	50	254.3	295.2	8.0	33.0	12.2
	3920851	F	Si/Al-DOX- VAR	<b>7</b> 5	255.7	296.8	8.5	30.8	14.4
9Ni-4Co(2	00)								
-	3920795	A	VAR-C-DOX	25	175.9	191,5	18.2	66.9	50.5
	E5565	В	AM-Si/Al- DOX	25	166.4	202.9	15, 5	<b>4</b> 6.5	20.3
	3920835	С	Si/Al-DOX- VAR	75	170.7	205.2	17.7	58,5	26.1
18Ni(250)									
Heat No.	3321290	G	AM-ISLVD	75	255.8	263.2	9.5	46.0	14.7
	3920864	н	VAR	75	273.1	280.5	9.2	45,3	16.7
18Ni(300)									
Heat No.	08749	J	VAR	75	283.7	287.9	9.?	45.9	16.1

(a) -65 F.

18NI STEELS AS DETERMINED IN ROOM-TEMPERATURE TESTS

					Transvers	2		
G <sub>nc</sub> , inlb/in. <sup>2</sup>	K <sub>nc</sub> (a), kai /In.	Fty, ksi	Ftu.	Elongation, percent in 2 in.	Reduction in Area, percent	Impact <sup>(a)</sup> , ft-1b	G <sub>ne</sub> , in -lb/in. <sup>2</sup>	K <sub>nc</sub> (a), lai √in.
91.1	56.4	220.2	261.6	11.0	38,2	18.2	84.1	58.1
94.5	45.4	210.7	263.3	9.0	24.2	14.4	74.6	50.5
99.0	54.7	211.1	262.4	11.7	<b>3</b> 6.0	27.6	85.5	<b>4</b> 8. <b>4</b>
74.1	49.5	243.7	281. 5	8.0	30.9	13.1	65.5	<b>4</b> 6. 5
53.4	42.0	253.6	293.7	6.0	16.4	13.2	45, 9	43. 9
32.7	26.2	254.1	296.2	8.0	28.4	13.0	40.3	36.4
300.9	99.8	178.3	192,8	17.5	61.9	<b>4</b> 3. 1	277.3	91.8
197.6	86.5	171.7	203.1	13.0	35.6	19.3	152.0	13 2
278.8	88.8	171.6	206.8	16.0	51.9	28, 1	190.7	84.3
127.1	<b>52.</b> 0	259.8	269.0	5.0	23.7	12.7	81.6	56. 5
187.5	56.5	276.7	284.3	6.0	32.0	12.7	156.6	64. 4
153,4	60.2	285.0	294.0	5, 5	26.3	11, 1	130.4	50, 6

TABLE 6. RELATIVE MENT OF MELT PRACTICE AT OPTIMUM FORGING REDUCTION BASED ON OPTIMUM AIR-MELT PROPERTIES FOR 18N1 MARAGING AND 9N1-4Co QUENCH AND TEMPER STEELS

	JNI-4CO COERCH FIRE TELL													40	
									Reduction	£	Сһагру		Fracture	Fracture Toughtees	
		Optimum Forging Reduction,	Yield Strength	rength	Tensile Strength	rength	Elongation I. T	E L	in Area	1 1	V-Notch L T		Temp,		ы
9	Melt Practice	percent	-2	H	. ]										
3			914 -4Co(250) Quench and Temper	)) Quenc	h and Tem	1	Steel (Bainitic Treatment)	litic Tre	arment)				•		
		S	1.00	1,00	1.00	1,00	1.00	1.00	1.90 1	1.00 1	1.00 1	1,00 F	FT(2) -65	1,00	1.00
ш	AM-Si/AI-DOX-DUVD	3 1	06	60	1,00	1.01	1,08	1,17	1, 10 1	1,45 1	1.45 1	1.22	RT -85	1,05	1, 14 0, 92
ŧτ	Si/A'-DOX-VAR	75	1, 00	ŝ	: :					55	1 47	1.28	RT	0,97	1, 13
Ð	VAR-C-DOX(5)	75	1.03	1,05	0.99	1.00	1.00	 	. EZ . T				-65	1.37	1.32
			98.11-4Co(250) Quench and Temper Alloy Sueel (Marrensitic Treatment)	SO) Querk	th and Ten	per Alloy	Sueel (Ma	rrensitic	Treatm	F)					
												•	Ę	00	1 00
шщС	A.MSi/Al-DOX-ESLVD Si/Al-DOX-VAR VAR-C-DOX <sup>(b)</sup>	50 7.5 7.5	1,00 0,99 0,95	1,00 1,00 0.96	1.00 1.00 0.95	1,00 1,01 0,96	1,00	1.00	1.00 1.28 1.31	1,00 1,69 1,86	1.00 1.18 1.15	1, 00 1, 15 1, 16	RT RT	0.61	0.88
1				971-40	921-4Co(200) Quench and Temrer Alloy Steel	ch and Te	orer Allo	y Steel							
					6	5	00	1,00	1.00	1.00	1.50	1.00	RT	. oc	1.00 2.00
60	A:M-Si/AI-DOX	22	1.00	1, 00	7° 30		•						-65	00.1	
. (	ci/Al-POX-VAR	75	1,03	1.69	1,01	10.4	1.26	1,46	1,14	1.23	30	 	*1 -65	1.05	1,33
~ ر	VAR-C-DOX(b)	25	1.06	1,04	0,94	6.93	4.	74	1, 18	1.34	2, 30	2.24	RT: -65	\$ 55 i ii	
;															
				181	18NK250) Maraging Steel	raginy Ste	ন্ত।				,	6	ž	1 00	
,	A 14 - PS   VT)	75	1, 40	1.00	1,00	1.00	1.00	1,00	1, 90	1.00	1,00	F. 69	-65	1.03	1.69
ا د		75	1.07	1.07	1.97	1,06	1,00	1.20	0.98	1.35	1.13	0.98	RT -55	1,48	1.29
I															

(a) RT = room ten perature. (b) Optimum melt practice for the grade and condition of heat treatment.

#### Fracture-Toughness Properties

The fracture toughness of the experimental steels was evaluated by the slow-bend specimen shown in Figure 1. The notch was located so that the fracture would propagate in the transverse direction. Before testing the notched beams, a fatigue crack was introduced at the base of the notch. At least 25,000 cycles were required by fatigue loading at an R factor of 0.5 to develop a crack 0.075-inch deep. Thus, the notch plus the crack reduced the thickness of the toughness specimen by 30 percent.

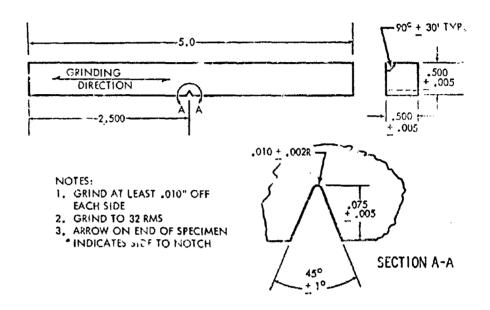


FIGURE 1. FRACTURE-TOUGHNESS SPECIMEN

The fracture-toughness tests were conducted on a small Universal testing machine at a loading rate of 1000 lb/min using a manual pacer. The load-deformation curves were recorded at a magnification of 250. A BLH-101M extensometer was used to measure the deflection of the specimen. The test fixture consisted of a apport with a 4-inch span and a pin-swiveled single-loading head.

To provide data for correlating the slope of the load-deflection curves with the depth of the crack at the initiation of crack propagation, a calibration curve was obtained for each alloy investigated. The calibration curves were determined on individual bars, within their elastic limits, with notch depths known to  $\pm 0.0005$  inch. The 45 degree notches had a radius of 0.010 inch and varied in depth from 0.100 to 0.275 inch in 0.025-inch increments.

From the load-deflection curves on the calibration bars, the spring constant "M" was determined for a particular crack depth. The "M" value is necessary for - plying the Irwin Equation\* for fracture toughness, which is:

$$G = \frac{1}{2} (P/B)^2 d/da (B/M)$$
 (1)

<sup>\*</sup>Irwin, G. R., et al., "Fracture Strengths Relative to Onset and Arrest of Crack Propagation", NU, ..eport 5222 (November 28, 1958).

To obtain the differential of the calibration curve for a particular crack depth, a, it is necessary to obtain the equation of the curve. The equations of the various calibration curves were determined by using a computer programmed for the Least Equares Method of regression analysis. The quadratic equations had the general form:

$$Y = AX^2 + BX + C (2)$$

The calibration equation coefficients for each material investigated in this Phase III of the study are given in Table 7. It shows that the coefficients are influenced by specimen orientation, the short-transverse grain direction having higher coefficients.

TABLE 7. CALIBRATION-EQUATION COEFFICIENTS FOR 1/2 x 1/2-INCH NOTCH-BEND FRACTURE-TOUGHNESS SPECIMENS - THREE POINT LOADING(a)

Material	Grain ' Direction	A x 10 <sup>-6</sup>	B > 10-6	C × 10 <sup>-6</sup>	Poisson's Ratio	Elastic Modulus (E) x 10 <sup>-6</sup> psi
9Ni-4Co(250) Bainite	Long. Trans	159, 996 182, 408	- 26. 006 - 34. 472	6, 28105 6, 87838	0.33	29.5
9Ni-4Co(250) Martensite	Long. Trans	178, 373 166, 913	-32, 6203 -28, 8485	6.74422 6.47366	0.33	29.5
9Ni-4Co(200)	Long. Trans	160,513 18',894	- 26. 8059 -37. 5429	6. 18458 6. 78538	0.33	29,5
18Ni(300)	Long. Trans	169. 888 171, 420	- 28, 3865 - 29, 9692	6,54070 6,68479	0. 30	27, 5
18Ni(250)	Long. Trans	186. 265 193. 612	- 34, 5949 - 37, 4985	7. 13333 7. 28112	0.30	26, 5

<sup>(</sup>a) Calibration curve form: B/M = Aa2 + "a + C; span to width ratio; 8:1.

A FORTRAN IV computer model was programmed to provide the following information about fracture-toughness characteristics:

- al Effective crack depth at initiation of crack propagation
- a. Effective crack depth at initiation of rapid fracture
- Gnc Crack-extension force, in.-lb/in. 2 (Determined from load at proportional limit and effective crack depth)
- \*G<sub>c</sub> Approximate critical crack extension force, in.-lb/in. <sup>2</sup> (Determined from maximum load and effective crack depth at onset of rapid failure)
- $K_{\rm he}$  Opening mode, stress-intensity factor, ksi  $\sqrt{in_e}$
- \* $K_c$  Approximate critical stress-intensity factor, ksi $\sqrt{in}$ .

The terms  $G_{nc}$  and  $K_{nc}$  were used instead of the customary symbols  $G_{Ic}$  and  $K_{Ic}$  to call attention to the uncertainties in determining the load at which crack instability first

occurred. That "pop-in" load is usually assumed to be the proportional limit. In this work, however, the pop-in loads were appreciably higher than the proportional limit. The data for two steels shown in Figure 2 illustrate this behavior. For consistency, the loads at the proportional limit were used in calculations. This means that  $G_{\rm nc}$  and  $K_{\rm nc}$  values were conservative; they were calculated from Irwin's formula, given below:

$$G_{nc} = 1/2 \left(\frac{P_{nc}}{B}\right)^2 d/da (B/M_A)$$
 (3)

and

$$K_{\rm nc} = \sqrt{\frac{E G_{\rm nc}}{1 - v^2}} ,$$

where

Pnc Load at proportional limit, lb

B Specimen width, in.

MA Initial load-deflection slope, lb/in.

V Poisson's ratio

E Elastic modulus, lb/in. x 10<sup>6</sup>

The data on fracture toughness are summarized in Tables 5 and 6. All the fracture-toughness values for the 9Ni-4Co(250) steels in the bainitic condition are lower than anticipated. Highest values were obtained with the VAR-C-DOX steel.

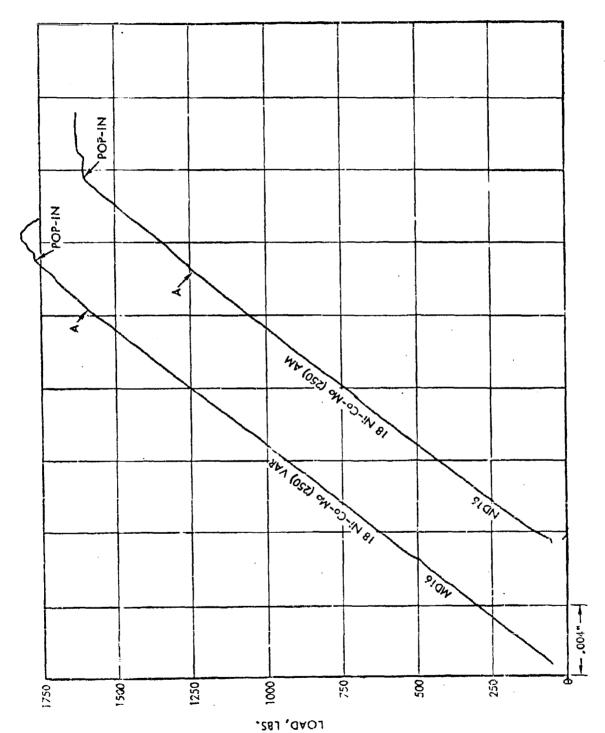
For the 9Ni-4Co(250) steels in the martensitic condition, the fracture-toughness values are also lower than those previously determined for this grade. Higher and more consistent values of fracture toughness were obtained in the VAR-C-DOX steel than in the Si/Al-DOX-VAR steel.

The fracture-toughness values for the 9Ni-4Co(200) steels also are lower than were previously determined for this grade by other investigators. It appears that the  $1/2 \times 1/2$ -inch slow-notch-bend specimens did not have a sufficiently large cross section to permit a valid evaluation of a material having a fracture toughness as high as this grade of steel. Consequently, the values reported are believed to have only qualitative, rather than quantitative significance.

Fracture-toughness values reported for the 18Ni-Co-Mo(250) steel produced by vacuum-arc remelting are much higher than those produced by air melting with the induction-stirred-ladle vacuum-degassing melting practice.

# Impact Properties

Impact tests were performed on standard V-Notch Charpy specimens at -65 F using a Richle Model PI-2 impact-testing machine that met the requirements for ASTM E23-64. The low-capacity (30 to 60 ft-lb) hammer was adequate for all steels except the 9Ni-4Co(200) steel (A), which required the intermediate-capacity (60 to 120 ft-lb) hammer.



LCAD-DEFLECTION CURVES OF 18 NI(250) FRACTURE-TOUGHNESS SPECIMENS EXHIBITING POP-IN BEYOND PROPORTIONAL LIMIT FIGURE 2.

Specimens were cooled to -65 F  $\pm 3$  F in a Dry Ice-acetone bath. The chilled specimens were handled with tongs chilled to -65 F. Temperature was monitored by means of a thermocouple attached to one of the specimens.

The impact data also are summarized in Tables 5 and 6. For the 9Ni-4Co(250) steel in the bainitic condition, impact properties greater than 15 ft-lb at -65 F were obtained in both the longitudinal and transverse directions for steels reduced 75 percent produced either by the Si/Al-DOX-VAR melting practice or the VAR-C-DOC practice. All steels of this grade in the martensitic condition had impact strengths lower than 15 ft-lb at -65 F.

Significant improvement (up to 150 percent) in impact properties at -65 F of the 9Ni-4Co(200) steel produced by VAR-C-DOX melting practice were noted over the same grade of steel made by the AM-Si/Al-DOX practice. This grade of steel had higher impact strengths than any of the steels studied in the present evaluation program.

None of the 18Ni maraging steels evaluated in this program had impact values at -65 F in the transverse direction as high as the minimum acceptable value of 15 ft-1b. In the longitudinal direction, steels produced by the VAR practice exceeded the 15-ft-1b minimum; the steel produced by the AM-ISLVD practice, reduced an equal amount (75 percent) in finish forging, was slightly below this minimum value.

### Metallographic Examination

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Macrosections, representing the complete cross section of each billet, were deep etched under the following conditions:

9Ni-4Co(200) and 250 Steels 50% HCl at 155 F for 1 hr

18Ni(250) and (300) maraging Steels swabbed with 20 cc HF  $\pm$  14 cc  $\rm H_2SO_4 \pm 5$  cc HNO<sub>3</sub>

Flow lines could not be effectively revealed by deep etching. No flow lines could be detected in the 9Ni-4Co steels reduced 25 percent in final finish forging; specimens reduced 50 and 75 percent revealed only faint flow lines. However, no evidence of alloy segregation, abnormal-inclusion content, or forging defects were noted in the macrosections.

The 18Ni maraging steels were much more difficult to macroetch, and what faint macrostructure was detected visually could not be reproduced on photographs. As with the 9Ni-4Co steels, no evidence for material defects such as banding, overheating, internal seams, and improper forging were found in the 18Ni maraging steels.

Although no metallurgical abnormalities or defects were uncovered from the macroetching studies, some defects were found in the 9Ni-4Co(250) steel "E" by a microstructural examination. While trying to explain the low fracture toughness in a specimen of this martensitic treated steel, Republic Heat 3321246, an abnormal microstructure containing banding and islands of white-etching ferrite at the grain boundaries was found in two different forged billets of this steel. However, this structure was far enough away from the fracture-toughness crack not to affect the test results.

The quality of the 18Ni-maraging-steel heats was judged to be very good on the basis of microstructural examinations. No be ding was evident, and only a very small number of inclusions were noted.

# Conclusions From Forging Reduction Studies

The overall results of the forging reduction studies show that the following melting practices, forging reductions, and finish forging temperatures provide the optimum processing for each material grade:

Material Grade	Melt Practice	Minimum Forging Reduction percent	Finish Forging Temperature, F
18Ni(300)	AV	<b>7</b> 5	1800-1850
18Ni(250)	VAR	<b>7</b> 5	1800-1850
9Ni-4Co(250)	VAR-C-DOX	75	1900-1950
9Ni-4Co(200)	VAR-C-DOX	25	1900-1950

# EVALUATION OF ROLLED-RING FORGINGS OF 18Ni (250) STEEL

An investigation of the variables of ring-forging production of the 18Ni(250) maraging steel was conducted by the Ladish Company. The objective of this program was to determine the influence of melting practice (air melt, vacuum degassed versus vacuum-are remelt) and of forging procedure on the metallurgical structure and mechanical properties of small-diameter ring forgings. The program consisted of producing and evaluating eight ring forgings made from air-melted steel and eight ring forgings made from vacuum-are-remelted steel. Variations in upset temperature, ring-rolling reduction and temperature, and heat treatment of the rings were investigated.

#### Materials

Two 15-1/4-inch round-cornered-square (RCS) as-forged billets of the 18Ni(250) maraging steel were supplied by Republic Steel Corporation. One billet had been forged from a 25 x 27-inch ingot of air-melted, vacuum-degassed Heat No. 3321290, and the other had been forged from a 24-inch-diameter ingot of vacuum-air-melted Heat No. 3920873 (parent Heat No. 3321290).

#### Materials Acceptance Tests

In addition to materials-capability-test results furnished by Republic Steel Corporation, a material-acceptance-test evaluation program was conducted by Ladish Company. The acceptance evaluation tests used at Ladish included chemical analyses, macroetch studies, microexaminations for cleanliness and grain size, mechanical property tests including tensile and precracked V-notch impact, magnetic particle and ultrasonic inspection, and gradient bar studies.

Chemical analyses indicated the billets to be uniform from end to end and that the composition agreed closely with that reported by Republic in Table 2. Results of deepetching studies of slices representing the top and bottom of each billet indicated that the VAR ingot contained fine uniform grain size and was free of porosity. These results were substantiated by results of ultrasonic inspection. Several surface cracks were found on the air-melted billet and center porosity was detected on the bottom slice. The evidence for porosity near the bottom of the air-melted billet was substantiated by ultrasonic inspections conducted both by Ladish and Republic Steel. The porosity extended about 4 feet from the bottom of the billet and six other locations of porosity were detected along the length of the billet. Reducing the size of the 15-1/4-inch RCS billet by forging to 12- and 10-inch RCS billets apparently healed the defects since no evidence of defects was found in the reduced redrawn billets.

Grain-size and cleanliness tests also were conducted as part of the material-acceptance program. At Ladish, both the as-received 15-1/5-inch RCS billet and the reforged 3-1/2-inch-square test bars were examined; at Republic, test bars forged to 5-1/2-inch squares had been studied. Thus, results from these two studies might be expected to vary to some extent. The reforging (reduction to 3-1/2-inch squares) at

Ladish refined the grains from predominantly ASTM 2 to predominantly ASTM 6 for both billets. The method used for rating cleanliness was Method A of ASTM designation E45. The method consists of visually comparing fields of most dense inclusions with a chart supplied for the standard. The ratings supplied by Republic Steel indicated significantly cleaner steels than those made by Ladish. Subsequently, micro-specimens were exchanged between the two laboratories and it was found that the sample-preparation technique and, hence, the appearance of the inclusions, could be improved by rinsing in plain tap water. However, not all of the difference in rating could be explained by the metallographic technique.

Tensile properties and precracked-Charpy V-notch impact properties were determined on samples of the 18Ni steel cut from the 3-1/2-inch reforged square test bars. The reduction in area by forging 75 percent corresponds to the maximum wall reduction planned for the seamless rolled-ring evaluation. Tensile and impact properties were determined both in the longitudinal and transverse directions. Tensile specimens were standard 0.252-inch-diameter specimens; impact specimens were 0.394-inch square. The reforged bar was solution annealed at 1650 F and then aged at 900 F for 3 hours. The data reported by Republic Steel were obtained on specimens cut from 5-1/4-inch-square bar annealed at 1500 F and then aged at 900 F for 3 hours. The data obtained at Ladish and those supplied by Republic Steel are summarized in Table 8. It is evident that the transverse yield strength is higher for the vacuum arc-remelted steel than for the air-melted steel. However, the transverse ductility values obtained at Ladish for the vacuum-arc-melted steel are significantly lower than those obtained on the air-melted steel. The W/A impact values of the vacuum-arc-remelted steel are significantly higher than those obtained on the air-melted steel.

As part of the acceptance tests, magnetic-particle inspection was used to obtain information on the degree of chemical segregation appearing as long stringers of austenite. The tests were made on sections of the 3-1/2-inch-square reforged test bar of each heat of steel. Bars for the tests were machined to 6-inch lengths 2-1/2 inches in diameter without steps. Excessive segregation was evident in both the air-melted and the vacuum-arc-remelted steel heats. No significant improvement was noted with regard to austenite segregation after a solution anneal at 1700 F followed by oil quenching.

# Forging Procedure

The 15-1/4-inch RCS billets were drawn down to 12- and 10-inch RCS stock by the following sequence:

- (1) Charge 15-1/4-inch RCS billet into 1250 F furnace and equalize
- (2) Raise temperature of furnace to 2250 F
- (3) Forge billet to 12-inch RCS and part off required amount of 12-inch stock and air cool
- (4) Reheat balance of 12-inch RCS billet to 2250 F, equalize, and torge down to 10-inch RCS; air cool.

MATERIAL-ACCEPTANCE MECHANICAL-PROPERTY TEST RESULTS FOR 18Ni(250) MARAGING STEELS TABLE 8.

	E							No test No	No. 3920873
Test Conditions and	Direction(a)	Ai	Me. ted	Air-Me ted Heat No. 3	3321290	Vacuum	Arc-nei	70173	1
				a, Ro	Room-Temperature	ature Tensile	ile Tests		
		Î L	H E	Elongation,	щ	Fty,	F <sub>tu</sub> , F	Elongation, percent	Reduction of Area, percent
		K81	ksi	percent	percent	Yeu		,	32.7
Tested by Republic;	H	254.1	262.7	9	34.5	250.6	258. l	Ó	
5-1/4-in, square		•							
solution treatment;									
900 F 3-hr age					,	•	25.4	10	51
	'n	242.8	254.0	10	51	240,4	256.9	17	53
Tested by Ladish;	ı	243, 4		10	44 ሪ ላ	244, 2	254. 2	ம	22
	₽ 6	229.9	254. 4 254. 6	æ <b>9</b>	27		256.4	ហ	<b>∞</b>
solution treatment;						,	•		
900 F. 3-111 &gc				(b) Pre	Precracked Charpy	rpy V-Not	V-Notch Impact	Testa	•
			6	Ĺ	CD W/A	T T	DBN	ធ	CD W/A
		Ļ	DBN	1	1		ر م	5, 59	0.025 577
Tested by Ledish; 3-1/2-in. square	11	0, 393	0,319 0,319 0,319	4, 22 4, 96 4, 45	0.030 443 0.030 521 0.030 467	900		5.24	0.020 532 0.020 645 Average = 585
after 1650 F, 1-hr		0.393		ì	Average = 477	۲.		•	
solution treatment. 900 F, 3-hr age		0, 393	0.319	2.20	0,040 23			9 2.82	0.040 307 0,035 330 0,040 315
	H H	0, 595	o o	; ;	11 0	279 0.39 259	o o	7. 30	11 0)

In order to obtain the maximum amount of data from a minimum amount of material, the following procedure was used:

- (1) The stock was upset at two different temperatures, 2250 and 1950 F
- (2) The final rings were rolled from two different temperatures, 2250 and 1950 F
- (3) The wall thicknesses were reduced by two different amounts, 50 and 75 percent, at each rolling temperature.

A temperature of 1950 F was selected for the intermediate hot-working operations, based on previous forgeability studies by Republic, on the maraging steel. Two methods of achieving low finishing temperatures were utilized:

- (a) Starting at a high temperature (2250 F) and ring rolling without reheating
- (b) Ring rolling at a low temperature (1950 F) and reheating several times.

Table 9 outlines the details of the forging program carried out at Ladish, and Table 10 gives the material requirements and the forging and seamless-ring rolling procedures that were used. After final ring rolling, the rings were sectioned and heat treated. One half of each ring was solution annealed at 1500 F; the second half was solution annealed at 1650 F. Both sections were aged at 900 F for 3 hours prior to testing.

TABLE 9. DETAILS OF FORGING PROCILAM FOR PRODUCING TWO DIAMETERS OF SEAMLESS RINGS FROM 18Ni(250) MARAGING STEELS AT LADISH

Draw-Down Operation:			d 15-1/4-Inch RCS d 10-Inch RCS	,
Upsetting Operation:	4 Ri Upset Appr 3:1 in I Furnace T	ngs roximately	4 Ri Upset Appr 3:1 in 1 Furnace Te	roximately Height;
Temperature Prior to Intermediate Rolling:	225	0 F	225	0 F
Temperature Prior to Final Rolling	1950 F	2250 F	1950 F	2250 F
Reduction A:	50 percent wall reduction (1 ring)	50 percent wall reduction (1 ring)	50 percent wall reduction (1 ring)	50 percent wall reduction (1 ring)
Reduction B:	75 percent wall reduction (1 ring)	75 percent wall reduction (1 ring)	75 percent wall reduction (1 ring)	75 percent wall reduction (1 ring)

# TABLE 10. MATERIAL REQUIREMENTS AND SCHEDULED FORGING AND SEAMLESS-RING-ROLLING PROCEDURES

# 50 Percent Wall Reduction

Stock Required: 10-inch RCS x 13-1/2 inches long

#### Forging Procedure:

Operation No. 1 Upset to 6 inches high and plug 5-inch-diameter hole

Operation No. 2 Ring roll and flatten to 12-inch inner diameter x 6-inch wall x 4 inches high

Operation No. 3 Ring roll and flatten (during rolling) to three-inch wall x 4 inches high

# 75 Percent Wall Reduction

Stock Required: 12-inch RCS x 16-7/16 inches long

#### Forging Procedure:

Operation No. 1 Upset to 4 inches high and plug 5-inch diameter hole

Operation No. 2 Ring roll and flatten to 6-1/2-inch inner Ciameter x 10-1/2-inch wall x 4 inches high

Operation No. 3 Ring roll and flatten (during rolling) to 2-7/8-inch wall x 4 inches high

# Mechanical Properties of Rolled Rings

Standard 0, 252-inch-diameter tensile-test specimens were used to obtain tensile data from the seamless rolled rings. Yield strength was determined by the 0.2 percent offset method. Precracked Charpy V-notched impact bars were 0.394-inch square. The impact bars were precracked in tension on a Manlabs Charpy precracker. Both tensile and impact specimens were tested as prescribed in Federal Test Method Standard 151a. Data were obtained for the axial, circumferential, and radial directions. Three specimens were tested for each specimen orientation.

The test results obtained are listed in Tables 11 through 15.

TABLE 11. YIELD STRENGTH DATA FOR 18NI STEEL(4)

0.2 Percent Offser

Material:		Nit-Melte		Air-Molted Alloy	d Alley			١	· Section (Section )		- (	- Prc-	Vacuum -Arc-Remelted Alloy	Í		
Percent Wall Reduction:			75			ડુ	ا	7		75				જ્ઞ		
Operting Temperature, F:		  -  -	2.5	2:2:50	1950	S	22	2250	1950	9	2250	00	1980	s) 8	28	\$250 \$300
Rolling Temperature, 8: Forging Serial Par.	1955	2250	3	4	1950	2250 16	1950	9555 82	1950	7	8 8	6 g	22	8	2 2	23
					Anne	a'ing Te	mperstur	Annesting Tempersture # 1500 F	n 1			٠				
<u>Test Dires to an</u> Circumerential	251.4 262.6 254.8	249.2 249.2 255.0	252.3 258.3 252.6	264.0 256.2 251.2	274. 4 257. 3 259. 9	24%.0 248,6 251.2	260.0 247.9 264.7	252.2 245.0 252.8	260, 6 256, 8 267, 4	272.4 251.0 270.5	257.2 264.0 263.0	263, 6 251, 2 251, 8	258.6 251.0 255.1	255.8 253.0 241.3	255.6 247.6 251.3	258.7 252.1 239.1
्रक्ष्फ्रहरू इस्ट्रोक्ट	251.0 255.4 250.6	250.6 246.8 256.2	257.7 253.5 253.4	250.2 253.2 248.6	256.3 256.6 246.6	252 0 . <sup>14</sup> .2 249, 6	250.4 252.0 253.2	244.0 248.8 244.6	260.0 260.6 263.1	271.3 263.9 253.6	262. 6 259. 0 262. 8	249.0 251.4 251.0	255. 0 252. 6 255. 5	252.0 251.2 251.0	255.7 258. \$ 260. 3	250, 1 250, 1 249, 1
Radial -? (P⊜! enty)																
Axial	254.0 250.8 264.2	248.2 251.4 260.2	253.6 252.8 253.6	257.6 254.2 250.2	262.3 241.3 356.7	251.8 252.2 251.0	254,8 247,9 251,6	247.5 245.2 246.0	261.3 261.2 280.6	263.0 264.4 257.1	264.5 263.3 263.9	248.8 250.6 261.1	249.6 250.6 258.4	252.6 251.6 252.4	259.2 260.0 265.2	249,6 243,6 252,5
					Ann	Annealing Temperature = 1650	mperatur	e = 1650	u.,							
Test Discotion Circumferential	250.2 346.6 245.9	247.0 244.4 240.8	242.9 244.2 248.6	244.6 241.2 244.2	250.4 247.1 247.9	243.6 247.9 247.2	252.4 252.6 254.6	253.2 252.6 252.2	253, 2 250, 2 252, 2	252. 3 241. 8 252. 0	252.6 249.2 247.0	249. 4 249. 8 254. 8	250 0 240.7 243.2	242.6 246.6 246.7	253.5 245.2 242.2	244.7 249.1 250.6
Radiei∗1	246.0 245.8 246.2	240, 2 243, 2 244, 0	241.2 244.2 242.4	241.4 241.6 240.2	253.4 243.6 234.6	246.2 240.2 245.0	254.2 254.6 250.2	258.0 254.6 247.0	250, 2 251, 2 256, 2	248. 2 249. 0 246. 6	249, 4 248, 6 246, 3	251. 8 246. 6 250. 2	247.4 244.0 347.4	241.6 241.8 252.1	248.5 299.6 241.1	250.1 253.1 248.6
Racial -2 (PCI only)																
Axial	244,8 247,8 243,2	243.4 247.4 242.5	248.6 243.2 238.5	235.7 241.4 240.9	245, <b>8</b> 243, 4 248, 6	243.4 243.4 207.9	256.6 239.8 233.8	250.2 233.9 246,8	252.2 251.6 251.0	252.6 249,4 252.2	250.6 251.2 256.2	250.6 250.0 251.8	247.4 249.8 245.6	243.7 241.6 241.6	240.3 245.2 249.6	246.1 245.2 244.6

(4) Specimens said at Sol Flores bous after solution annealing.

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TABLE 12. ULTIMATE-TENSILE-STRENGTH DATA FOR 18Ni STEEL<sup>(4)</sup>

Marcrial:				Air-Melted Alloy	ed Alloy						Vacu	um - Arc -f	Vacuum - Arc - Remetted Alloy	Alloy		
Percent Wall Reduction:			75			20	0			75					98	
Upsetting Temperature, F:	1950	S		2250	1950	S	2	2256	81	1950	22	2250		1950	2,	2250
Rolling Temperature, F. Forging Serial No.:	1950	2250	1950	2250	1950	225 <b>0</b> 18	1950	2250	1950	2250	1950 8	2250 9	1950	2250	1930	22.50
					V	nealing 1	emperati	Annealing Temperature = 1500 F	<u> </u>							
Test Direction Circumferential	265.3 263.9 266.0	260.3 259.7 264.6	260.8 264.6 262.7	273. 4 265. 0 263. 7	282.9 263.8 268.2	259.3 259.9 262.3	264.8 258.6 272.7	261.0 255.6 261.6	268. 3 262. 8 273. 7	277.2 260.6 275.3	264.6 271.5 271.8	270.8 260.6 262.3	268.5 265.0 266.5	264.0 259.6 261.5	268.5 251.6 264.0	266.8 264.7 261.6
Radis1-1	262.3 265.7 261.8	261. 5 257. 7 257. 8	265. 6 265. 2 262. 3	259.0 261.4 259.4	269.6 266.2 258.6	257.7 263.3 260.5	260.0 261.6 262.3	257.2 259.4 252.6	268.4 268.7 270.8	274.1 272.7 261.7	267.3 269.5 269.6	257.4 256.6 256.8	262.4 264.6 276.5	260.4 260.2 259.4	257.7 265.7 265.8	262.6 262.6 262.7
Radial -2 (PCI only)																
אַע ה	264.0 264.5 271.7	257.4 263.0 269.9	257.6 265.7 261.7	266.8 263.5 260.7	271.4 262.4 266.8	260.6 262.9 259.6	264.0 259.4 263.2	260.0 250.2 257.2	272.6 272.0 270.7	271. 5 272. 2 264. 4	27?.2 269.7 270.3	260.8 261.1 269.8	264.6 277.6 265.0	260.6 260.6 260.6	270.7 270.0 278.7	262.6 262.6 263.6
					\[ \begin{array}{c} \equiv \left\right\rig	ncaling I	emperati	Annealing Temperature = 1650	띩	•						
Test Direction Circumferential	260.6 257.9 257.4	280.6 25.0 255.6	256.7 254.6 258.6	254.6 252.8 255.0	261.6 257.8 258.6	254.4 258.4 258.0	259.7 261.6 264.9	261.7 263.4 261.8	262.0 259.1 262.6	262.1 258.6 260.9	262.3 258.6 257.0	258.6 259.6 261.8	258.6 255.6 257.6	255.6 2.5.6 258.6	263.6 260.6 261.6	261.6 259.7 261.6
Radial - 1	258.0 257.6 259.0	254.8 256.4 252.2	251.2 255.4 253.6	251.6 252.2 242.2	259. 4 254. 0 246. 6	258.2 251.4 256.2	261.8 262.4 259.4	266.0 253.6 254.1	260.2 260.1 261.9	257.4 257.4 258.9	260.2 256.6 257.2	259.8 257.0 260.0	255.0 254.6 256.6	254.6 258.0 258.6	261.6 260.6 261.8	261.6 262.6 263.6
Radial -: (PCI only)																
Axial	257.6 259.0 258.2	256.6 280.7 255.0	257.9 254.0 256.3	252. 2 253. 8 253. 8	255.8 257.8 260.2	249.8 253.2 255.8	265.8 250.6 245.6	261.8 241.6 257.6	262.3 261.8 261.1	262.3 259.8 262.3	259.8 259.6 264.1	259.9 260.3 262.0	258.0 257.2 256.6	258.6 .58.6 255.6	252.6 231.6 204.6	261.6 261.6 253.6
As Section in asset of Shi F for I have after solution annealing.	F for 3 ho	Her after	solution a	n.iealing.												

TABLE 11. PERCENT ELONGATION TEST DATA FOR 18N1 STEEL<sup>(4)</sup>

				Air-Melted Alloy	1 Alloy						- 1	n-Arc-he	Vacuum-Arc-Kemelted Alloy	) 5		
Material:						S				75			100		500	5
Percent Wall Reduction:		5			٤	501	İ	2250	2	1950	2250	S	881		200	2
Upsetting Temperature, F. Rolling Temperature, F. Caning Serial No.	1950	2250	1950	2250	1950	18	1950	2250	1950	2250	1950 8	9	22	22.50	200	8 8
911951					<b>∢</b> 1	mealing	Tempera	Amcaling Temperature = 1500 F	90 F							
Test Direction Circumferential	8 8	10 5 10	10 10 9	3 9	<b>∞</b> ⊱ o	<b>∞</b> ∞ ∞	9 01 6	01 8 e	2 2 2	<b>01</b> 00 <b>0</b>	00 00 10	10 10 10	<b>ထ</b> ထ တ	r- 00 r- 0	<b>കക</b> അ ∨	⊬n eo eo ∈
Radial-1	φφn	& 6. T	t~ 64	ର <b>ଦ</b> ର	აბ <b>ად</b>	404	୯୩୯୯	F 23 F	Ø <del>4 4</del>	<b>9</b> - 9	61 63 FG	ፍመል	<b>୬</b> ପୋଟ	ကယ	വ വര	- <del></del>
Radial -2 (PCI only)																
Axial	၈ယေးက	H E &	61 VS	E- 4-0		6 2 Annealing	8 3 5 g Temper	6 8 8 8 2 2 5 2 Annealing Temperature # 1650 F	6 6 650 F	c- 00 ♥	m 04 10	<b>+</b> 40 57	4 থ 4	4 4 0	* 4 10	ro, c. co
Test Direction Circamferential	01 9 01	ន្ទន	9 9 6	8 8 8	<b>எ ச ச</b>	æ ∞ <b>∞</b>	8 01 8	கைசு	221	91 91 91 91	01 9 8	9 01 9	<b>த</b> வ வ	10 10 10	222 '	gneu aó é
Redial - 1	<b>80 80</b> 01	<b>∞</b>	40	4 5	9 Q	<b>କ</b> ପାରା	9 13 49	900	L to a	t- t- 0	വ വ	พรก	r <b>u 4</b>		⊷ es es	ુ ના <b>પ</b> ક
Radial -2 (PCI only)							,	•	•	đ	4	•77	•	<b>(</b> -	<b>4</b> 0	•
Axial	<b>φ </b>	s 8	ø €4	o * '	<b>★</b> 67 (		<b>ගෙ</b> ෆ ◀	<b>∞</b> ⊣ ⋈	- 6 -	, <b>4</b> 00		, c. æ	<b>я Ф</b>	4 13	en en	• *
	•	-	က		<b>P</b>	7	,	•								

(a) specimens aged at 900 F for 3 bours after solution annealing.

TABLE 14. PERCENT-REDUCTION-OF-AREA TEST DATA FOR 18NI STEEL (4)

Automating Temperature = 1850 E   1950   1	6 6 7 7 7 7 8 8 8 8 8 8 8 8 8 8 8 8 8 8			2250 2 2 2550 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
Annealing Temperature = 1500 F  44	다 않 <b>않 않</b> 성 성 성 성 성	·	22 4.4.4.4.3.3.5.5.5.5.6.6.4.8	64 84 85 85 85 85 85 85 85 85 85 85 85 85 85
14	48 46 52 50 38 49 47 45 49 7 33 26 21 5 22 23 2 18	• •	25 6 48 43 12 23 6 48 43 12 23 24 43 12 24 24 24 24 24 24 24 24 24 24 24 24 24	34 48 3 5 4 4 8 1 18 4 4 8 1 18 4 4 8 1 18 4 18 18 18 18 18 18 18 18 18 18 18 18 18
11   20	50 38 43 45 45 45 45 45 45 45 45 45 45 45 45 45		1. ± \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$ \$	8 4 4 8 8 4 8 8 1 4 8 8 1 1 1 1 1 1 1 1
41 20 7 30 26 31 14 15 17 30 25 31 40 45 46 42 37 40 35 35 31 40 35 31 14 15 17 30 25 31 31 32 31 31 31 31 31 31 31 31 31 31 31 31 31	47 45 49 47 33 26 21 5 22 23 2 18		\$ • 55 ° E E	3 8 4 4 4 4 18
11   20	21 5 22 21 5 22 23 2 18		25. 5 23. 5 24. 5 25. 6	4. e.
1	21 5 22 . 23 . 24 . 25			3 4 4 8 E
8	. 23 2 18		្ន នូង	D & 4 4 8
8 27 37 50 34 16 15 12 20 15 20 15 11 11 11 11 11 11 11 11 11 11 11 11			33	4 4 81
8 27 37 50 34 34 16 15 12 20 15 8 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			33	₹ ₹ ₹
2 6 12 2 13 26 14 31 9 20 16 16 11 3 18 5 26 19 36 45 21 13 27 11 13 27 11 13 27 11 13 27 11 14 29 20 11 15 21 13 27 11 14 29 20 21 26 18 24 45 46 11 24 24 20 20 20 20 20 20 20 20 20 20 20 20 20	37 50 34		8	18
Annealing Temperature = 1650 F  43	12 2 13	•		18
Annealing Temperature = 1650 F  43	18 5 26		64	
43         40         42         40         54         53         53         53         51         46         43         40         34         46         45         46         46         48         40         45         46         46         46         48         49         45         46<		Anne		
45 36 46 39 52 51 46 48 49 45 46 46 49 45 46 46 49 45 46 46 49 48 49 45 46 46 46 49 48 49 45 46 46 46 46 46 46 46 46 46 46 46 46 46	42 40 54	·	8	<b>.</b>
45 34 45 46 11 49 48 49 45 40 40 40 40 40 40 40 40 40 40 40 40 40	46 39 52		<b>\$</b>	8
27     14     29     21     36     33     26     18     34     25     27       30     8     7     7     27     31     24     20     20     28     18       38     8     25     4     24     24     21     23     17     21       16     2     41     26     34     41     18     25     22     32     40       43     4     13     4     35     36     50     44     34     22     17	45 46 11		41	51
30 8 7 7 27 31 24 20 20 22 18 38 8 25 4 24 24 21 23 17 21 20 20 18 18 18 18 18 18 18 19 19 19 19 19 19 19 19 19 19 19 19 19	23 24 35		40	ď
38 8 25 4 24 21 23 11 21 20 11 21 20 11 16 2 41 26 34 41 18 25 22 32 40 15 3 16 25 19 29 33 12 13 24 24 15 3 3 3 5 5 4 34 34 22 17	7 7 27		35	9 00
16 2 41 26 34 41 18 · 25 22 32 40 15 3 16 25 19 29 30 12 13 24 43 4 13 4 35 36 50 44 34 22 17	25 4 24		64	Ĵ
16     2     41     26     34     41     18     25     22     32     40       15     3     16     25     19     29     33     12     13     24       43     4     13     4     35     36     50     44     34     22     17				
15 3 16 25 19 29 33 12 13 24 42 4 13 4 35 36 50 44 34 22 17	76 56	9	Ş	•
43 4 13 4 35 36 50 44 34 22 17	16 25	9 1	200	2.
	13 4 35	; <del>;</del>		7 -

(a) specimens aged at 900 F for 3 bours after solution annealing.

Material:				Air -Melted Alloy	d Alloy							n-Arc-Re	Vacuum -Arc -Remelted Alloy			
Fercent Wall Reduction:		7	75	į		\$				2						
Upsetting Temperature, F:	1950	8	2	2250	1950	95	22	2250	1950	S	الثر		1950	ا	- i	22.50
Rolling Temperature, F: Forging Serial No.:	1950	22.50	3 8	22.55 4	158	18	1950	3250	1950	7	1950 8	22£ J	1950 22	2230	1350	22.50
c.c.																
					•	Annealing	Temper	Annealing Temperature = 1500 F	500 F							
Test Direction	8	133	364	617	383	989	6	340	603	\$27	593	83.	\$08	327	878	4.35
Circumierennau	36.	100	204	385	374	988	376	386	635	205	624	455	464	336	£4	76 65
	\$23	571	388	383	354	298	382	333	587	<b>438</b>	613	461	473	345	613	<b>9</b>
Radial-1	315	<b>4</b> 39	536	251	308	569	317	305	368	304	295	300	311	285	294	3.5
T THIRD	342	86	782	259	300	274	383	295	367	305	334	265	285	285	276	345
	392	371	234	245	569	270	274	549	332	307	287	279	274	283	326	311
Calaibad	247	312	600	298	269	<b>5</b>	368	25.	276	260	220	252	235	276	247	311
(A) (A)	285	<b>3</b>	332	780	252	257	797	258	270	281	330	240	262	8	242	3.9
(Les out)	9 20 20	378	<b>1</b> 88	314	181	360	420	274	279	24€	244	267	242	252	235	2 []
	;	;	Č	ć	6	,	ç	c	3	77	394	4.85	411	428	331	ឌ្ណ
Axial	380	407	385	281	<b>0</b>	361	245	3	3	11.	• •	3	1 6	0.5	71.6	20.0
	367	456	<b>8</b> 60	335	279	307	8	339	392	427	ş	391	22.5	ers.	916	5
	363	577	485	301	329	300	342	265	425	<del>1</del> 05	613	322	988	330	380	Ž:
					•	Annealing Temperature # 1650 F	Temper	sture # 1	550 F							
Ten Direction															;	:
Circumferential	505	571	487	442	878 8	344	356	368	į	226	635	487	<b>1</b> 63	376	455	3
) )	8	929	495	450	342	337	349	346	<b>\$69</b>	Z	577	25	99	346	513	354
	513	515	423	430	46.8	325	337	363	739	532	575	661	477	374	538	Š.
r Parking	346	2	555	265	808	277	302	520	443	35	315	232	317	276	314	278
	968	323	407	273	338	293	302	963 23	416	360	314	243	322	302	314	4 64
	395	36	358	<b>5</b> 62	327	277	287	297	439	390	351	307	333	283	369	3
o-Teibed	346	443	176	358	303	221	269	437	375	388	256	307	288	569	2967	280
Saulai - 2	2 0	2.2	385	666	φ. •	247	255	569	392	338	255	314	272	280	27.5	282
(rei ouy)	385	<b>4</b> 10	374	305	2,2	227	25	271	.330	315	314	285	272	290	35. 4	26.
	906	410	37.1	376	202	282	346	346	516	487	352	450	409	387	264	327
ANIAI	236	408	415	312	938	292	38	306	£38	44	634	462	344	322	387	398
	385	391	694	S	371	274	282	335	613	527	387	445	420	452	389	311
	; } }	:														

TABLE 15. PRECRACICED CHARPY V-NOTCHED IMPACT DATA FOR 18NI STEEL<sup>(A)</sup>

(4) specimens aged at 900 F for 3 hours after solution annealing.

# Statistical Analyses Procedures

The tensi: and impact data obtained were analyzed statistically by three nithods of statistical analysis. These are the analysis of variance (ANOVA), cumulative frequency-distribution curves, and linear-correlation analysis.

Analysis of Variance. The data in Tables 11 through 15 initially were analyzed by the analysis-of-variance method. This method involves studying all of the variability or variance found in the data and partitioning it off in such a way that it is possible to distinguish the variability associated with each variable or combination of variables.

The data were evaluated using a four-factor  $2 \times 2 \times 3 \times 8$  "analysis of variance". Following are the four main variables and the subvariables that were evaluated:

V, ≈ Melting practice

Air melting Vacuum-arc remelting

V<sub>2</sub> ≈ Forging practice

Forged at 2250 F

Rolled at 2250 F - 50 percent reduction Rolled at 2250 F - 75 percent reduction Rolled at 1950 F - 50 percent reduction Rolled at 1950 F - 75 percent reduction

Forged at 1950 F

Rolled at 2250 F - 50 percent reduction Rolled at 2250 F - 75 percent reduction Rolled at 1950 F - 50 percent reduction Rolled at 1950 F - 75 percent reduction

V<sub>3</sub> = Heat treatment

Solution annealed at 1500 F and aged at 900 F

Solution annealed at 1650 F and aged at 900 F

 $V_{\underline{A}} = Testing direction$ 

Tensile

Axial Circumferential Redial

Impact (Precracked Charpy V-Notch)

Axial
Circumferential
Radial - notch open to tace of ring
Radial - notch open to cross section face of ring.

The test data were utilized in statistical program on an IBM 7040 computer to analyze for significance between the tain effects and the interactions.

Details on the computer analy and the data are given in Appendix D of the Final Report dated 30 September, 1965, a moved by Ladish for Republic Steel Corporation under Air Force Prime Contract and second-order interactions, the incluence exerted by an experimental variable was considered significant and requireg additional examination only if it showed significance at the 1 percent confidence level in two of the three sets of experimental data.

Cumulative Frequency-Distribution Curves. The magnitude of the influence exerted on the mechanical properties by the main variables and the interactions was determined by the use of the cumulative frequency distribution curve. This tool can readily provide the relationship between any value and its confidence level.

Linear Correlation Analysis. In conducting the linear correlation analysis, the following procedure was implemented:

- (1) Only circumferential properties were used because the analysis of variance indicated testing direction had a significant influence on some of the mechanical properties and because cumulative frequency distribution curves showed that the best data distributions came from circumferentially oriented test results.
- (2) The correlation analysis was completed by analyzing 96 test results each of tensile yield, elongation, reduction of area, and fracture-toughness properties for linear correlation with each other. This analysis showed very good linear correlation between circumferential properties of yield and ultimate tensile strengths and between percent elongation and reduction of area, and precracked Charpy values.
- (3) In order to determine whether melting process or heat treatment affected the correlation analysis, the 96 circumferential yield and ultimatestrength test results were divided by melting process into two groups of 48 values each and the analysis was rerun. The correlation factor for the air-melted group was 0.94574 and for the vacuum-arc-remelted group, 0.90248. The 96 results were then re-arrayed by solution-annealing temperature into two groups of 48 values each, and the analysis was again rerun. Correlation factor for the 1500 F solution-annealing temperature was 0.91635, and for the 1650 F group, 0.79891.

# Results of Statistical Analysis

Tensile Data. The analysis of variance indicated that only the heat-treatment and melting practice had significant influence on both the yield and tensile strength. In addition, forging practice exhibited significant influence on the yield strength but had no significant influence on the tensile strength. Testing direction was the only variable that showed a significant influence on the ductility as measured by elongation and reduction of area. None of the first- and second-order interactions showed significant

influence on either the yield strength, tensile strength, elongation, or reduction of area on the basis of the analysis of variance study.

Cumulative frequency distribution curves were used to determine the numerical extent of the variables on properties. Figure 3 shows the cumulative frequency distribution curves in which the influence of solution-annealing temperature on both yield and tensile strength is indicated. The figure shows that both the tensile- and yield-strength medians for the steels solution annealed at 1500 F are 5000 psi higher than those for the steels annealed at 1650 F.

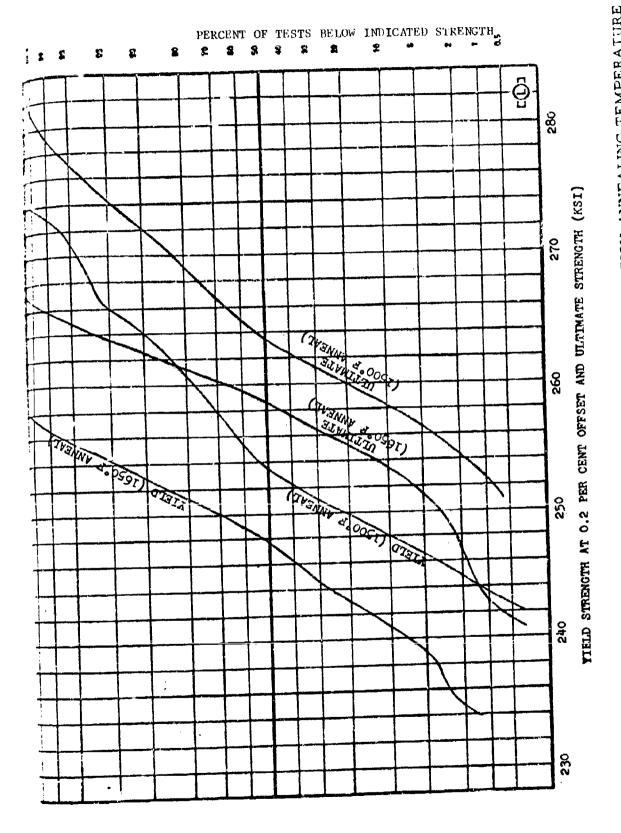
Figure 4 shows the cumulative frequency distribution curves to the tensile and yield strengths of air melted versus vacuum-arc remelted. It can be seen that about 2000-psi higher yield and tensile strength was obtained to the mediums with the vacuum-arc-remelted steel than with the air-melted steel.

Cumulative frequency distribution curves showing the influence of forging practice on the yield and tensile strengths did not single out any particular torging practice as being superior to the other practices.

Cumulative frequency distributions curves showing the influence of testing direction on elongation and reduction of area are shown in Figure 5. The circumferential specimens showed 4.5 percent greater elongation and 23 percent greater reduction of area for the medians than the transverse (axial and radial) directions. It may also be noted from Figure 5 that the confidence level is about 98 percent for achieving 5 percent elongation and 25 percent reduction of area in the circumferential direction, but at this confidence level the transverse ductility is virtually nil. Even if one restricts consideration to the vacuum arc-remelted heat, the 98 percent confidence level is only 13 percent reduction of area and 2 percent elongation in the transverse directions. The data on both reduction of area and elongation presented in Tables 13 and 14 suggest that if ductility is of primary concern, superior transverse properties may be obtained by using vacuum-arc-remelted steel and solution annealing the material at 1650 F, although statistically the data are of dubious significance.

Fracture Toughness. The analysis-of-variance method of statistical analysis indicated that melting practice, forging practice, and testing direction all had significant influence on fracture toughness as measured by the precracked Charpy V-notch impact test results. Two first-order interactions, melting practice with testing direction and forging practice with testing direction, had significant influence on fracture toughness.

Cumulative frequency distribution curves were used to determine the extent of the effects. Figure 6 shows the curve of precracked Charpy V-notch impact test results by testing direction. The curves and supporting histograms indicate that the test results are not normally distributed and, in fact, represent two different populations or a skewed distribution. The reasons for this abnormality are not known and the extremely low values of the 18Ni-(250) maraging steel, both air and vacuum-arc remelted, should be thoroughly studied before being selected for critical fracture toughness applications. The "end-of-fiber" orientation (R<sub>2</sub>) was consistently lower than the "side-splitting-fiber" orientation (R<sub>1</sub>).



STRENGTH OF 18-N; STEEL AS INFLUENCED BY SOLUTION-ANNEALING TEMPERATURE Cumulative frequency distribution curve. FIGURE 3.

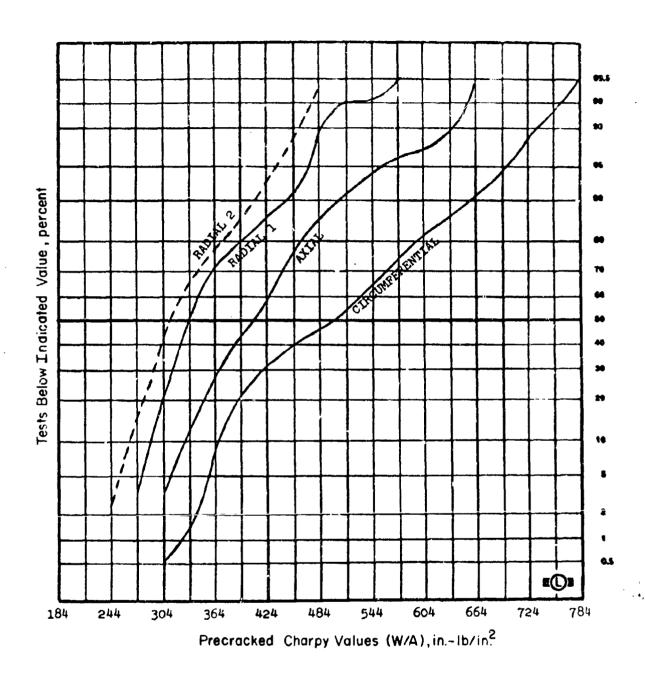


FIGURE 6. FRACTURE TOUGHNESS OF 18Ni STEEL AS INFLUENCED BY TESTING DIRECTION

Cumulative Frequency Distribution Curve.

The cumulative frequency distribution curves in Figure 7 show the influence of testing direction and melting practice on fracture toughness. Higher fracture toughness is evident for vacuum-arc-remelted steel for both the circumferential and axial test direction. The results for the radial "end-of-fiber" (R<sub>2</sub>) orientation show higher fracture toughness for the air-melted steel than for the vacuum-arc-remelted steel. For the "side-splitting-fiber" orientation, R<sub>1</sub>, there is little difference between the two steels. Highest values of fracture toughness were obtained for the circumferential orientation and lowest for the radial orientation.

A plot of the eight cumulative frequency distribution curves showing the influence of forging practice resulted in a distinct separation of three practices having fracture-toughness values greater than the other five. The plot is shown in Figure 8. The three forging practices that showed the highest values of fracture toughness are:

- (1) 75 percent wall reduction, 1950 F upsetting temperature, and 2250 F rolling temperature
- (2) 75 percent wall reduction and 1950 F upsetting and rolling temperatures
- (3) 75 percent wall reduction, 2250 upsetting, and 1950 F rolling temperature.

Analysis of Forging Practice. The forging practices utilized in this program were selected around the three following criteria:

- (1) Upsetting the stock at 1950 or 2250 F, followed by preliminary rolling at 2250 F
- (2) Accomplishing the final wall reduction by heating to 2250 F and working over a range of lower temperatures without reheating, or by restricting the heating temperature to 1950 F and completing the working using several reheats
- (3) Reducing the wall thickness either 50 or 75 percent in the final operation.

Since the analysis of variance was set up to evaluate a forging practice as a total entity, an analysis of the eight combinations of forging practice cumulative frequency diagrams for each of the above three criteria also was made. The analysis of circumferential test results showed that upsetting temperature had no significant effect on fracture toughness, tensile strength, or ductility.

The rolling practice produced a significant difference in means of values for fracture toughness and tensile reduction of area. Figure 9 shows that rolling over a falling temperature range from 2250 F resulted in a median circumferential fracture toughness W/A value of 490 in. -1b/in. 2 compared with a median value of 415 fm-1b/in. 2 for rolling at 1950 F. Similarly, Figure 10 shows a median value of 47 percent reduction in area for rolling over a falling temperature range from 2250 F, compared with 44 percent for rolling at 1950 F. Thus, the method of rolling from 2250 F over a decreasing temperature range is the preferred method for the 18Ni(250) maraging steel.

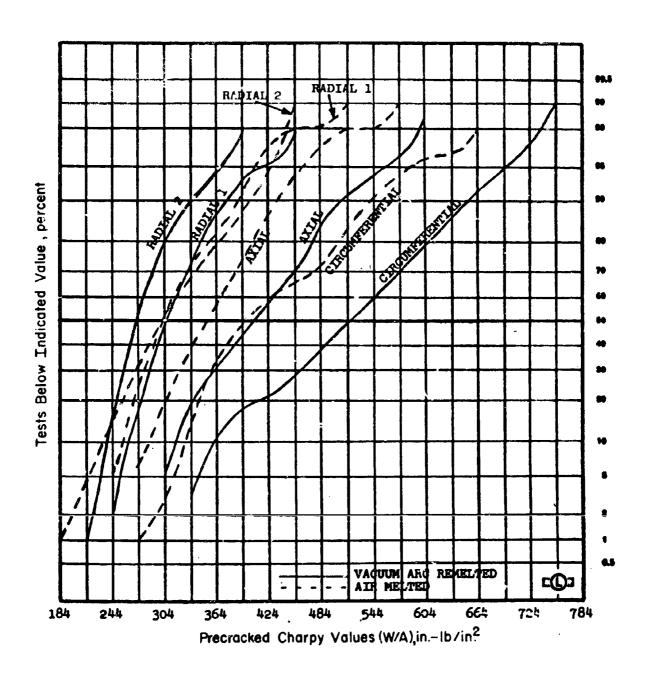


FIGURE 7. FRACTURE TOUGHNESS OF 18Ni STEEL AS INFLUENCED BY TESTING DIRECTION AND MELTING PROCESS

Cumulative Frequency Distantion Curve.

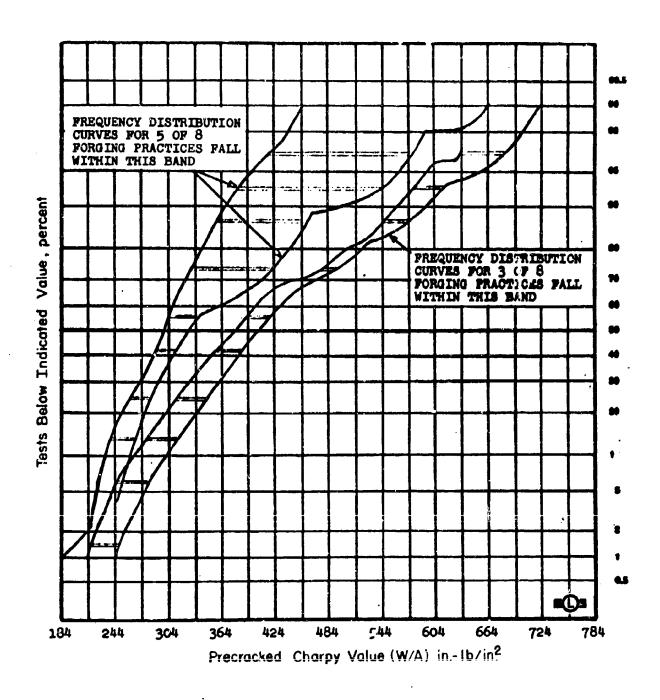


FIGURE 8. FRACTURE TOUGHNESS OF 18Ni STEEL AS INFLUENCED BY FORGING PRACTICE

Cumulative Frequency Distribution Curve.

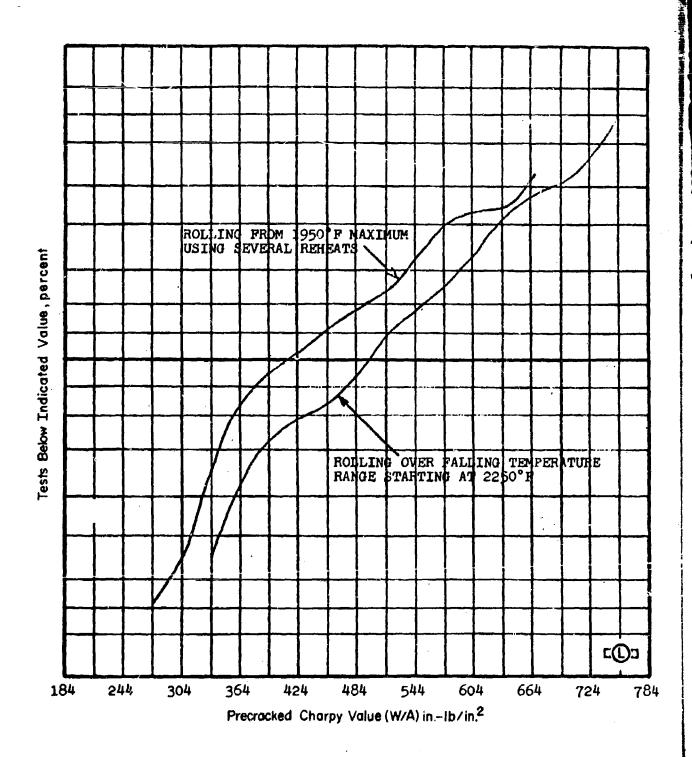


FIGURE 9. FRACTURE TOUGHNESS OF 18Ni STEEL AS INFLUENCED BY ROLLING TEMPERATURE

Circumferential Specimens,

Cumulative Frequency Distribution Curve.

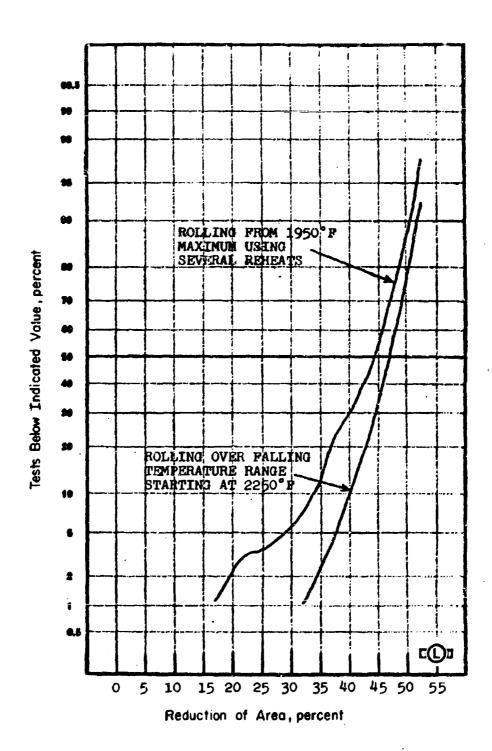


FIGURE 10. REDUCTION OF AREA VALUES FOR CIRCUMFERENTIAL SPECIMENS
OF 18Ni STEEL AS INFLUENCED BY ROLLING TEMPERATURE
Cumulative Frequency Distribution Curves.

The severity of plastic deformation in the final ring-rolling operation had a significant effect on the ductility and fracture-toughness, but not on the strength levels. Figure 11 shows the cumulative frequency distribution curves in which the effect of wall reduction on circumferential fracture toughness is plotted. The difference at the median is about 180 in, -1b/in, 2. Although the difference at the median is less when axial and radial as well as circumferential values of fracture toughness are used in the curves, the 75 percent wall-thickness reductions produce higher average values than the 50 percent reductions.

Figure 12 shows the cumulative frequency distribution curves for the effect of wall-thickness reduction on ductility. At the medians, the elongation is about 1.5 percent and the reduction of area about 5 percent greater for a 75 percent wall-thickness reduction than for a 50 percent reduction. An extreme shift occurs in the 75 percent curves at the lower range.

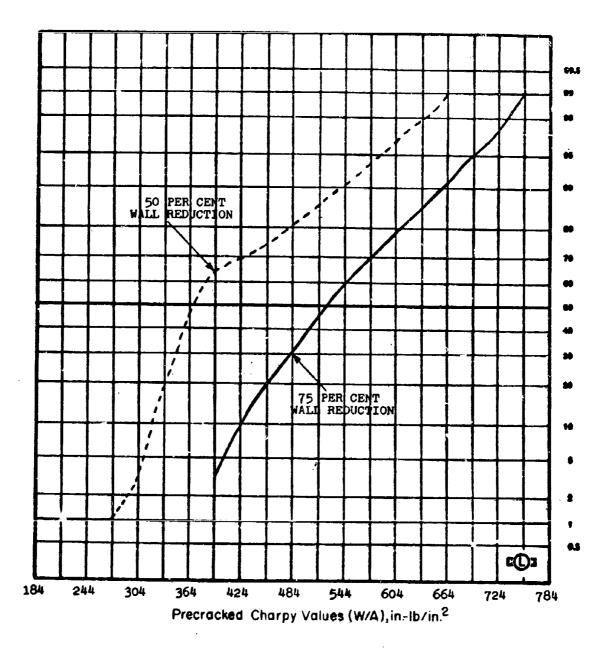
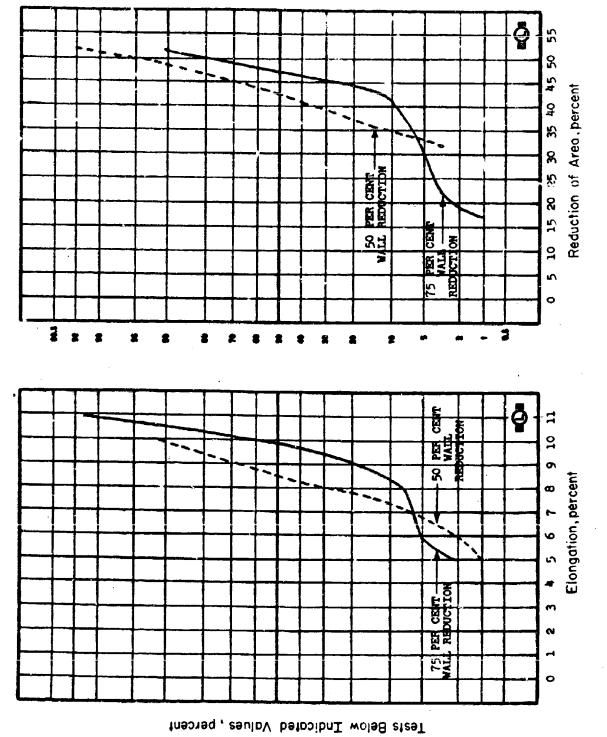


FIGURE 11. CIRCUMFERENTIAL FRACTURE TOUGHNESS AS INFLUENCED BY THE AMOUNT OF WALL REDUCTION AT FINAL ROLLING Cumulative Frequency Distribution Curve.



CIRCUMFERENTIAL DUCTILITY OF 18N; STEEL AS INFLUENCED BY THE AMOUNT OF WALL REDUCTION AT FINAL ROLLING Cumulative Frequency Distribution Curve, FIGURE 12.

# EVALUATION OF RING-ROLLED FORGINGS OF 18Ni(300) AND 9Ni-4Co STEEL

### Prior History

Various properties of 18Ni maraging and 9Ni-4Co alloy steel samples taken from ring-rolled forgings were evaluated by Aerojet-General Corporation. The processing history of the 21-in. ID x 24-in. OD x 5-in. rings is indicated in Table 16. Tests on four forgings of each grade of material permitted determinations of the uniformity of mechanical and metallurgical properties as a function of location and crientation within a torging and between forgings and heats.

Each forging was inspected ultrasonically using Sonoray Model 50 C equipment with a 3/4-inch-diameter 5.0-mc ZT Branson Transducer. The contact-scanning technique, with a water column, was employed in all instances. An A4130 alloy-steel test standard was used and the scanning sensitivity determined by calibrating to a 3/64-inch-diameter flat-bottomed hole at a 5-inch metal path. No defects were found in the maraging or 9Ni-4Co(250) steel forgings. Inspection of Ring No. 4, of the 9Ni-4Co(200) steel, indicated two defects located 3/4 inch from the surface. One indication was equivalent to a 3/64-inch and the other to a 2/64-inch deep flat-bottomed hole. Neither was found by sectioning.

TABLE 16, MELTING(a) AND PROCESSING HISTORY OF RING-ROLLED FORGINGS

		·		Ring	Rolling Conditi	ons
Heat N	lumber	Melting		Temperature	Temperature	Reduction,
Original	Final	Practice	Grade	at Start, F	at Finish, F	percent
		18	Ni Maras	ing Steel		
3321284	3920878	VAR	300	2025	1825	60
	3920879	VAR	300	2025	1825	<del>6</del> 0
		91	Ni-4Co A	lloy Steel		
3321262	3921091	VAR-C-DOX	250	2025	1925	60
	3951502	VAR-C-DOX	250	2025	1925	60
3311846	3921032	VAR-C-DOX	200	2025	1925	60
	3931021	VAR-C-DOX	200	2025	1925	60

<sup>(</sup>a) The heats were produced by Republic Steel Corporation. The original 70-ton heats were melted in air and cast as 9500-lb ingots. That material was vacuum-arc remelted and cast as 8500-lb ingots. The temperatures listed are in the middle of 50 F ranges. The dimensions of the rolled rings were: 21-in. ID x 24-in. OD x 5 in.

Figure 13 illustrates the practice followed in sectioning most of the rings. The precracked Charpy specimens were taken from the 9Ni-4Co(200) steel at the locations shown for slow-notch-bend specimens; that is, on either side of the metallographic specimens.

Table 17 shows that, within the limits of accuracy of analytical methods employed, forgings of the same grade had identical compositions. This good control of composition

FIGURE 13. LOCATIONS OF SPECIMENS USED FOR EVALUATING PROPERTIES OF RING-ROLLED FORGINGS

might be expected because the two heats representing each grade came from the same original air-melted heat. All chemistry contents fit the appropriate specifications except for the nickel and cobalt levels of the 9Ni-4Co(200) steel. For that material, the nickel content exceeded the maximum limit of 8.0 percent and the chromium content was below the minimum limit of 0.35 percent mentioned in the specification.

TABLE 17. CHEMICAL COMPOSITIONS OF RING-ROLLED FORGINGS

	Range	s in Composition, p	percent(a)
Element	18Ni(300)	9Ni-4Co(250)	9Ni-4Co(200)
С	0.02/0.03	0.41/0.44	0,25/0,28
Mn	0,06/0,07	6,21/0,29	0.23/0.24
Si	0.07/0.09	0.03/0.06	0.031/0.034
P	0.008/0.009	0.005/0.010	0.002/0.003
S	0.003/0.005	0.006/0.006	0.008/0.010
Ni	18.11/18.62	7.93/7.95	8.45/8.56
Co	8. <b>6</b> 6/8.85	3.56/4.59	4.03/4.12
Mo	4.73/4.86	0.23/0.27	0.38/0.40
Al	0.16/0.16	0.05/0.06	0.04/0.04
Ti	0.70/0.79		<del>**</del>
Cr		0.23/0.24	0.33/0.34
v		0.09/0.09	0.08/0.08

<sup>(</sup>a) For two forgings from two vacuum-arc remelted heats of each grade. Aluminum content is not specified for the 9Ni-4Ge alloy steel.

#### Tensile Properties

The tensile tests were made on 0.25-in.-diameter x 1.0-in.-gage-length bars meeting ASTM specifications, except that button heads were used. The longitudinal ar transverse specimens represent hoop and axial directions in the rings, respectively. The data are summarized in Table 18.

All specimens of the 18Ni maraging steel met the tensile property requirements Specification RSC-AF-1 established for this program. The lower strengths and better ductilities of Ring 7 compared to Ring 5, which came from the same heat, are attribut to unintentional variations in forging practice. The average tensile properties did not differ significantly between heats or with specimen orientation. The data suggest that differences of about 15 ksi may be expected among forgings of 18Ni(300) steel produced from VAR heats made from the same melting stock and processed by the same nominal practices.

Ring 13 had lower yield and ultimate strength than anticipated for 9Ni-4Co(250) steel. Apparently this resulted from a mistake in heat treatment, because the other three rings had normal properties when tested after a second heat treatment. The marmum difference in yield strengths of the 36 specimens from those rings amounted to 10 ksi. The average yield and ultimate strengths of the 9Ni-4Co(250) rings were 224. I and 264.7 ksi, respectively. The tensile properties did not differ significantly between forgings or heats or with specimen orientation.

TABLE 18, MECHANICAL PROPERTIES OF SPECIMENS FROM ROLLED-RING FORCINGS(4)

Heat Number	Ring Number	Specimen Orientation	Ultimate Strength, ksi	0.2 Percent Offset Yield Strength, kei	Elongation, percent (1-in. gage)	Reduction in Area, percent	X <sub>nc</sub> Value, ksi √in,
			18Ni Maraging	Steel, Grade 300	VAR(b)	.,	
3920879	5	L	293, 5	286.5	5, 8	21.4	57
	-	Ť	296, 5	288,5	4.5	16,7	57
		sT				~-	53
3920879	7	L	280.5	272.5	7.0	24.9	58
- • •		Ť	283.0	273,5	6.9	25, 8	58
		ST		••		4. <del></del>	54
3920878	8	L	285, 5	279.5	6.0	21,8	61
-,	- ,	T	288.0	280.5	6.2	21,5	59
	•	TZ	'				56
3920878	10	L	287,2	278.7	5.5	21,2	58
0,200.0	••	T	286, 1	273.5	4.9	18.9	- 59
		S'T					57
		•	9Ni-4Co Alloy	Steel, Grade 250	VAR(c)		
3921691	13(d)	L	227,5	189.0	13.5	54, 0	66
		T	.227.5	192.0	13.0	54, 5	68
•		ST					68
3951502	11	L	264.0	224.5	12.0	52.5	70
0,000		T	264.5	223,5	13,0	47.5	72
		ST		·		·	68
3951502	12	L	264.5	224,0	12,5	52.5	74
-,51200		T '	265, 5	223.0	13,5	48.5	73
		ST					73
3921091	14	L	265, 5	226.0	12.5	47.5	65
3,210,1		Ť	265.0	224.0	12.5	48.0	69
		ST					68
			9Ni-4Co Alloy	Steel, Grade 200	VAR(6)		
3931021	1	L	196.5	187.5	18.0	63,5	
2,31021	-	T	197.0	187,5	17.0	60,5	
		ST	~ ~		••	***	<b>&amp; E</b>
3931021	2	L	197.3 -	186.9	17.2	62.5	
		T	198.7	188,3	. 17.3	55.0	/ <del></del> .
		ST					
3921032	, <b>3</b>	L	200.5	191.3	17.5	62,5	
3,2	-	Ť	201.0	192.3	17.3	60.0	
		ST					
3921032	4	L	199.5	190.5	17.8	62.5	
-,	•	Ť	1 9.0	190,5	17.0	59.5	
		ST					

<sup>(</sup>a) Each tensile value is the average of six observations; triplicate specimens from Location B1 and B2 in Figure 1. Each Knc value for longitudinal and transverse directions is based on 10 to 12 observations; the short-transverse values were usually based on 4 specimens.

<sup>(</sup>b) Solution annealed, 1650 F for 1 hour and aged at 925 F for 3 hours.
(c) Bainitic heat treatment; normalized 1650 F, austenitized 1475 F for 1 hour, salt quenched and held at 475 F for 6 hours.

<sup>(</sup>d) These specimens were, apparently, heat treated improperly. Therefore, the specimens from Rings 11, 12, and 14 were given a second heat treatment.

<sup>(</sup>e) Normalized at 1650 F for 1 hour. austenitized for 1 hour at 1550 F, quenched to room temperature in oil, double tempered at 1000 F, 2 hours for each treatment.

The forgings of 9Ni-4Co(200) steel had remarkably uniform tensile properties; neither specimen orientation nor location had any influence on strength of ductility. Although the difference in strengths of the two heats — about 4 ksi — is statistically significiant, it is of no engineering importance.

### Fracture Toughness

Slow-bend notched-beam tests were performed on specimens from the 18Ni maraging steel and the 9Ni-4Co(250) steel. The  $K_{\rm nc}$  values given in Table 18 were obtained on 0.500 x 0.500 x 5.0-inch bars by procedures described in a previous section. Equations developed by Bueckner\* were also employed for calculating  $K_{\rm Ic}$  values. The Beuckner analysis was preferred by Aerojet-General investigators because the values were less affected by experimental problems. Since the  $K_{\rm Ic}$  values approximated the  $K_{\rm nc}$  values and showed the same trends, they are omitted from this report.

The plane-strain fracture-toughness values of the 18Ni(300) specimens ranged from 53 to 61 ksi  $\sqrt{in}$ . The values were lower for short-transverse specimens, but the differences between longitudinal and transverse specimens were not significant. The values are better than those obtained for another lot in an earlier study. Fracture usually occurred at the maximum load, with no indication of slow crack growth. This behavior was considered an indication of lower toughness and a smaller critical-defect size compared to lower-strength steels.

Specimens of 9Ni-4Co(250) steel exhibited a proportional limit during testing followed by continued deflection before fracture. This is considered to be an indication that this steel has better plane-strain fracture properties than the 18Ni(300) maraging steel, but the yield strength was also lower. The  $K_{nc}$  values were significantly better; they ranged from 65 to 74 ksi  $\sqrt{\text{in}}$ . The fracture-toughness properties appear to be equivalent in all directions within the forged rings of this steel.

The slow-bend notched-beam tests did not detect differences in plane-strain toughness between forgings from different heats.

Precracked Charpy impact specimens were used to evaluate the fracture-toughness of the 9Ni-4Co(200) steel forgings, since a prior study had shown that valid plane-strain fracture-toughness values could not be obtained on this material using a 1/2 x 1/2-inch bend specimen. Standard V-notch Charpy bars, containing a fatigue crack approximately 0.05-in. deep below the machined notch, were used for the studies. The W/A values (breaking energy/cross-sectional area) are believed to represent an integration of the properties controlling the initiation (plane-strain conditions) and propagation of fracture. The results of the tests are summarized in Table 19.

The precracked Charpy specimens indicated that the fracture toughness of the 9Ni-4Co(200) steel was highest in the longitudinal direction and lowest in the short-transverse direction. Anisotropy of this kind is easier to detect in softer steels. The W/A values did not vary significantly as a function of location within a ring or between forgings.

<sup>\*</sup>Bueckner, H. F., "The Stres: Concentration of a Notched Bar in Bending", Large Steam Turbine-Generation Department, General Electric Company. Data folders dated June 14 and 29, 1957.

TABLE 19. PRECRACKED CHARPY IMPACT VALUES FOR 9Ni-4Co(200) SAMPLES FROM RING-ROLLED FORGINGS(a)

Heat	Ring	Specimen	Average 0,2 percent Yield	Impact Value (W/A) at Location Indicated, inlb/in. <sup>2</sup>					n
Number	Number	Orientation	Strength, ksi	Al	ΑZ	Bl	* B3	A4	B2
3931021	1	L	187.5	2718	2333	2775	2456	2712	2436
		T	187.5	1571	1610	1628	1750	1733	1730
		ST				1564			1658
3931021	2	L	186.9	2410	2344	2830	2260	2396	2641
		T	188.3	1733	1647	1569	1442	1705	1587
		ST				1539			1817
3921032	3	L	191.3	2883	2869	2524	2470	2572	2597
		T	192.3	2203	2020	2210	2241	2216	2258
		ST				2031			2184
3921032	4	L	190.5	2566	2370	2461	2531	2513	2419
		T	190.5	1816	1676	2227	1929	1852	2088
		ST				1875			1918

<sup>(</sup>a) The values are averages for three determinations. The specimens were austenitized for 1 hour at 1550 F, quenched in oil to room temperature, double tempered at 1000 F, 2 hours for each treatment.

### Metallographic Examinations

With one exception, all forgings met the inclusion rating requirements specified for this program. One location (E-2) in Ring 10 of the 18Ni(300) maraging steel received a rating of 3.5 for "thin" nitride inclusions, compared to a requirement of 3.0. The inclusion ratings were independent of specimen location and orientation.

All specimens exhibited microstructures typical of their grade and heat treatment. No significant variations were apparent between heats or specimen locations. The ASTM grain size of the 18Ni(300) maraging steel was judged to be mainly 3 to 5. This relatively coarse grain size may have resulted from an undesirably high finishing temperature when the rings were rolled. Both grades of 9Ni-4Co steel had grain sizes of ASTM 8 or finer. Their macrostructures showed some evidence of irregular grain flow. This suggests that the reduction by ring rolling was insufficient to reorient the patterns established by prior working.

#### EVALUATION OF CLOSED-DIE FORGINGS

This part of the report covers the evaluation of mechanical properties by General Dynamics/Fort Worth on 20 forgings. The forgings, which weighed 38 pounds, were made in dies used for producing the Wing Pivot Forward Support Bracket for the F-111 aircraft. The part (12B751-7-E) has section thicknesses ranging from 1/2 inch to 5 inches. In production operations, a considerable amount of metal is removed from all surfaces of the forging.

### Material

The compositions of the steels as determined on the forgings are listed in Table 20. For all practical purposes they fell within the specified ranges; the variations between forgings from the same heat and from different heats were small. The phosphorus, sulfur, and chromium contents of the 18Ni(250) air-melted vacuum-degassed heat "N" (No. 332190) and of the vacuum-arc-remelted heat "M" were almost identic. The latter heat (No. 3920873) contained slightly less silicon and manganese but more carbon. It had been produced by remelting stock from air-melted, vacuum-degassed Heat No. 3321290, which provided material for comparison.

The forgings were produced by Ladish-Pacific on a 12,000-lb drop hammer from 3-3/4-inch round-cornered square billets rolled by Republic Steel Corporation. The 9Ni-4Co alloy steel had been prepared by vacuum-arc-remelting of two air-melted heats. The 18Ni maraging steels represented four heats, one air melt and three vacuum-arc remelts. As-received by General Dynamics, the 18Ni steel had a hardness of 293 BHN. It had been solution annealed for 1 hour at 1635 F and then air cooled. The 9Ni-4Co steel had been normalized after holding 1 hour at 1650 F, then tempered for 2 hours at 1200 F; the hardness was 331 BHN.

The forgings passed ultrasonic inspection for internal defects. Surface defects detected by Magnaflux inspection were shallow enough to be removed by normal machining. Some small laps were present on all forgings and the surfaces were rough, presumably because of scaling during forging. All of the 9Ni-4Co forgings and those from 18Ni(300) heat Nos. 3920879 and 3920878 completely filled the die cavity. Incomplete die filling at the extreme outer edge of the 4-1/2-inch-wide section was noted in the 18Ni(250) forgings from Heat No. 3920873 and in three of the forgings from the 18Ni(250) air-melted heat.

Figure 14 is a schematic sketch showing the size and shape of the forgings and the location of test specimens. The 33-1/2-inch dimension was considered to be the longitudinal grain direction. The direction of the grain in Specimens 5 to 17 in forgings A, B, D, and E was arbitrarily considered the short-transverse direction because those specimens were perpendicular to the parting plane of the forging. Metallographic studies of grain flow, however, showed that the so-called short-transverse direction should have been called the long-transverse direction.

TABLE 20. CHEMICAL COMPOSITION OF STEEL FORGINGS

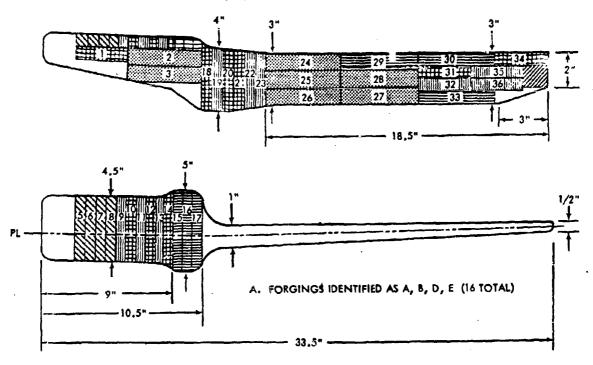
1

Material	O	Mn	Ь	S	Si	Ö	ž	Si Cr Ni Co	Mo	Ti	Ψ	۸
18NK(300)												
Vacuum Arc Remelt												
Heat No. 3920879												
KA 101	. 016	.11	800.	900.	. 12	8.	18.40	8.78	4.92	٤.	.11	:
KB 102	. 020	.11	600.	900.	. 12	8.	18.40	8.82	4.95	٠ 20	.11	:
Heat No. 3920878												
KC 201	610.	. 12	.007	.003	. 10	01.	18.44	8.84	4.92	17.	.11	:
KD 202	610.	. 12	.007	.004	01.	. 10	18.42	8.80	4.92	٥.	.11	:
KE 203	.018	. 12	800.	<b>7</b> 00.	. 10	01.	18.40	8.80	4.91	.7:	.11	;
Specified	.03 max	. 10 max	. 010 max	.010 max	Xar 101.	:	18-19	8.5-9.5	4.7-5.2	69.	.0515	:
13NW250)			•									
Vacuum-Arc Remelt												
Heat No. 3920873							i,					
MA 501	.015	8.	. 007	. 003	.07	. 10	17.86	7.88	4.97	.45	.11	:
MB 502	.015	8.	-001	.003	٠o.	91.	17.86	7.84	4.9	.45	.11	;
MC 503	. 015	.10	900.	.003	.07	. 10	17.88	7.86	4.98	<b>.</b>	.11	:
MD SO4	.017	. 10	900.	.003	٠٥٠	. 10	17.82	7.82	5.01	<del>9</del> .	.11	1
ME 505	.020	. 10	.005	.003	.07	요.	17.82	7.82	5,01	<del>4</del> .	.11	;
Air Melt												
Heat No. 3321290				•		3						
NA 601	.015	.11	.005	.003	٠٥.	. 30	17.86	7.80	5.00	.46	.11	:
709 <b>92</b>	.015	.11	900.	.003	.07	. 10	17.88	7.80	4.98	₹.	.11	:
NC 603	.015	.11	. 005	.003	.07	. 10	17.88	7.80	4.98	₹.	. 10	;
ND 604	.016	. 11	. 009	<b>*</b> 00 <b>*</b>	8.	. 10	17.86	7.80	4.95	<b>.</b>	.11	;
NE 605	<b>.</b> 01 <b>4</b>	.11	900.	<b>5</b> 00.	80.	. 10	17.88	7.80	₹.93	<b>4</b> .	.11	:
Specified	. 03 max	. 10 max	. Э10 тах	.010 max	. 10 max	:	17.5-18.5	7,5-8,5	4.7-5.2	.a. 5	.0515	:
9Ni-4Co(250)					÷ '							
Vacuum-Arc Remelt												
Heat No. 3951502				•								
36 14	<b>‡</b>	. 15	900.	600.	.01	.31	7.82	3.96	.30	:	:	8
1.8 305	. 425	.15	900.	600	. 01	.30	7.82	<b>4.</b> 96	.31	:	:	8
Heat No. 3921091												
10 401	. 425	. 14	. 005	600.	.01	œ.	7.82	3.98	.31	:	:	<b>3</b> .
F02	.431	.15	. 805	600.	.01	œ.	7.80	3,96	.30	:	:	\$
LE . 403	.427	. 15	900.	.008	.00	.30	7. 32	3.98	.30	;	:	<b>8</b> 0.
Caenified	07	40	240.010	210		30.00	7-0 7	5 P-5 &	50.00	;	;	26-15

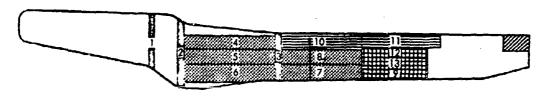
Chemical analysis performed by Republic Steel, Canton, Ohio. Letters (c.g., KA) are General Dynamics' Code; numbers (e.g., 101) are Ladish-Pacific code.

69

# A. FORGINGS IDENTIFIED AS A, B, D, E (16 TOTAL)



### E. FORGINGS IDENTIFIED AS C (4 TOTAL)



### NOMENCLATURE

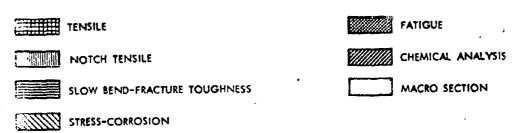


FIGURE 14. LOCATION OF TEST SPECIMENS IN FORGINGS

### Machining and Heat Treatment of Specimens

The tensile and fatigue specimens of 18Ni steel were finish machined prior to aging. The stress corrosion and fracture-toughness specimens were ground prior to aging. The notches of the fatigue specimens were polished, after aging, with levigated alumina on a Dacron cord; the stress-corrosion specimens were polished with 500-grit emery paper and vapor honed.

The 9Ni-4Co samples were rough machined oversize and finish machined after heat treatment. All notching was done after heat treatment; the finishing procedures were identical to those used for the maraging steels.

The impact specimens were taken from failed fracture-toughness specimens and notched in the same direction.

The 18Ni specimens were aged for 3 hours at 925 F. The heating and cooling times ranged from 1.25 to 1.82 and 2 to 2.5 hours, respectively. All specimens from the forgings of each alloy were heat treated together. In the case of steels "K" and "M", the maximum temperature in the outer retort was higher than desired, but tensile tests indicated that the properties were not affected significantly.

The heat treatment for developing a bainitic structure in the 9Ni-4Co steel was:

- (1) Austenitize at 1475 F for 1 hour
- (2) Quench into molten salt at 475 ±10 F and hold for 6 hours
- (3) Air cool with no further heating.

In the first attempt to heat treat the samples, the temperature of the salt bath reached 505 F. This resulted in slack quenching and undesirably low properties, so most of the specimens were heat treated again. Those specimens were tempered for 2 hours at 850 F before re-austenitizing. It was established that the second heat treatment, performed to eliminate the effects of slack quenching, did not affect the tensile properties adversely.

# Tensile Test

Tensile and notched-tensile tests were conducted on standard 0.252-inch-diameter specimens and on the notched-tensile specimen shown in Figure 15. Subsize specimens were necessitated by the size of the forging and the number of specimens needed for the program. They were attached to 18-inch-long draw bars, spherically seated in the crossheads to minimize bending. The loading rate was 5000 lb/min to the yield point; yield strengths were calculated by the 0.2 percent offset method. The tensile properties are summarized in Tables 21 and 22.

TABLE 21. TENSILE PROPERTIES OF 18Ni (300) VACUUM ARC REMELTED STEEL PORGING CODE "K"(a)

Forging	Elongation in 4D, percent	Reduction in Area, percent	F <sub>ty</sub> , kui	F <sub>tu</sub> , ksi	F <sub>ntu</sub> , ksi	Notched Ratio(b)
		Longi	tudinal Specime	ems		•
A	8.3	41.2	288.4	295.7	308.0	1,04
В	7.7	38.6	285,6	292, 1	302.6	1.04
D	8.7	41.8	288.4	295.5	312, 7	1.06
E	7.7	35,7	285.9	293.3	298.5	1,02
		Trans	verse Specimen	4		
A	7.3	32.8	280.3	289,2	290,5	1.00
R	7.7	32,3	284.3	292.4	274,6	0.94
D	8.7	37,6	283.5	291.4	279.7	0.96
E	7.8	34,6	281.9	290.0	288,6	1,00
		Short-T	ransverse Specin	nens		
A	8.0	<b>33.</b> 6	282.2	289.2	279.8	0.97
В	6.0	25.7	281.6	288.8	254, 8	0.88
D	6.5	25.3	282.9	291.2	299.3	1.03
Ē	7.7	30.8	284.2	290.9	252.0	0,87

<sup>(</sup>a) All values are averages of triplicate determinations. Forgings A & B from Heat 3920879; Forgings C, D & E from Heat 3920878 specimen number, as indicated in Figure B were as follows:

	Unnotei. d	Notched
Longitudinal	1, 31, 34	32, 25, 36
Transverse	18, 20, 21	19, 22, 23
Short transverse	12, 12, 14	9, 11, 13

<sup>(</sup>b) The K<sub>1</sub> values were 6.9 for all notched specimens except A22 and D0 which were 5.7 and 6.2 respectively.

TABLE 22. TENSILE PROPERTIES OF FORGINGS HEAT TREATED TO ULTIMATE STRENGTHS RANGING FROM 260 TO 280 KS(\*a)

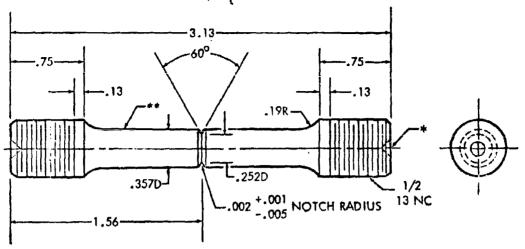
Specimen Orientation	Forging	Elongation in 4D, percent	Reduction in Area, percent	F <sub>ty</sub> , ksi	F <sub>tu</sub> , ksi	F <sub>ntu</sub> , ksi	Notched Ratio <sup>(b)</sup>
		18Ni (250) Vacu	um-Arc Remelt Forging	Code "M"			
Longitudinal	A	9.3	46.4	257.2	265.3	330,2	1,24
•	В	9.0	43.0	257.9	264.2	331.1	1,25
	D	9.5	42.5	260.4	266.5	323.2	1.21
	E	9.8	45.9	259.7>	<b>266.</b> 7	<b>331.</b> 6	1.24
Transverse	A	8.8	44.5	253.2	260.7	319.5	1,23
	В	8.3	42,4	25 <b>2.</b> 8	259.6	316.5	1.22
	D	9.3	43.4	254.9	262.2	321.4	1.23
	E	9.0	<b>4</b> 3.7	255.8	262.9	324.8	1.24
Short Transverse	A	8.5	41.4	255.2	261.3	330.9	1,27
	В	8.7	38.8	254.7	260.4	326.5	1,25
	D	8.5	38.7	257.7	263.7	320.3	1.21
	E	9.0	38.6	255.6	261.6	331.4	1.27
		18Ni (250	Air-Melted Forging Co	de "N"			•
Longitudinal	A	9.3	38.2	267.0	274.6	329.7	1.20
•	В	9.0	38.0	267.1	274.6	313.8	1.14
•	D	8.8	38.9	267.4	275.5	316.2	1. 15
	E	7.8	37.8	270.7	278.1	329.0	1.18
Transverse		7.7	32,7	262.0	271.3	304.2	1.12
	В	(10.0)	(41.6)	(259.5)	(268, 1)	309.2	1, 18
	D	8.8	37.1	263.6	271.4	280.2	1.03
	E	7.2	29.0	262.1	270, 1	305, 1	1.13
Short Transverse	A	5,3	16.0	261.9	270.3	<b>239.</b> 0	0.89
	В	6.0	22.6	260.8	268.8	289.9	1.08
	D	5,2	14.2	257.3	266.4	270.5	1.02
	E	5.7	22,0	262.2	270.7	261,9	0.97
•		9Ni-4Co (250) \	/acuum-Arc Remelt For	zing Code "L"			
Longitudinal	A	(13.0)	(57.5)	(229.7)	(267.0)	346.3	1.30
· ·	В	(13.0)	(53.2)	(229.6)	(265.8)	332,6	1.25
	D	(11.7)	(52.2)	(235.4)	(267.9)	334.8	1.25
	E	(14.0)	(56.3)	(231,6)	(268. 1)	336.8	1.26
Transverse	A	11.7	<b>4</b> 6.4	230.6	265.0	329.6	1.25
	В	11,5	47.0	231.1	264,6	338.8	1.28
	D	11.7	<b>4</b> 8,6	229.9	265.1	326.3	1.23
	E	13.0	49.1	230.1	266,8	326.6	1.22
Shor. Transverse	A	11.0	40.7	230.0	265, 8	326.7	1.23
	В	11.3	41.7	229.3	1266.6	333.5	1.25
•	D	11,8	41.8	229.5	265.6	<b>330.</b> 6	1.24
	E	12.0	44,0	231.9	266.7	327.1	1,23

<sup>(</sup>a) Values are averages of triplicate determinations, except those in parentheses, which were based on one or two tests. Heat identifications are given in Table 16. Specimen locations are shown in Figure 14 and listed in footnote of Table 21.

<sup>(</sup>b) The "K<sub>t</sub>" values were 5.7 for Group "N" and 6.9 for Group "M". In Group "L", the "K<sub>t</sub>" values averaged 6.3 for longitudinal specimens, 6.5 for transverse specimens, and 6.0 for short-transverse specimens.

<sup>(</sup>c) Data for Code "L" were obtain i on specimens given a double heat treatment.

# NOTCH TENSILE SPECIMEN, K. = 6.9



\*CENTER FOR TURNING & GRINDING (TYP.)

\*\*SURFACE TO BE CONCENTRIC WITHIN .001 T.I.R.

#### FIGURE 15. TENSILE-TEST SPECIMENS

Table 21 gives the tensile properties of the 18Ni steel as heat treated to an ultimate strength approximating 291 ksi; as is typical of maraging steels, the ratios of yield to ultimate strength exceeded 0.95. The strengths were approximately 4000 psi higher in the longitudinal than in the transverse directions. The elongation and reduction in area values were also poorer for the transverse specimens, and the differences attributable to orientation are statistically significant. The ductility values are not considered unusual for this strength level.

The notched specimens were designed to provide a stress concentration factor  $(K_t)$  of 6.9. The notched strengths are good for steel tested at such a high strength level. Only 2 of 12 longitudinal specimens broke at nominal stresses lower than the unnotched ultimate strength. As anticipated from the effect of orientation on ductility, the other specimens had lower notched-strength ratios. The average ratios of notched to ultimate strength were 0.97 and 0.94 for transverse and short-transverse specimens, respectively. The three short-transverse specimens from forging "E" had notchultimate ratios below 0.9.

The tensile properties of samples from forgings with strengths ranging from 260 to 280 ksi are listed in Table 22. In general, the reproducibility of data among samples from different forgings was good. The air=melted 18Ni(250) steel had a higher strength and poorer ductility values than its vacuum-remelted counterpart. The uniformity was better for the latter material and comparable to that exhibited by the arc-remelted samples tested at the higher strength level. In both materials, the ductility and notched-ultimate ratios were better for specimens oriented in the transverse than in the short-transverse direction. Longitudinal specimens exhibited the best properties. In all but two cases, both for air-melted steel, the average notched-ultimate strength ratios exceeded 1.0.

The data for the 9Ni-4Co steels are difficult to assess for reproducibility because of the necessity for a second heat treatment. However, the spread in values seems reasonably small. The yield-ultimate strength ratio of this steel, about 0.8, is less than that for the maraging steels. Regardless of specimen orientation, all tests showed the material to possess good notched strengths; the ratios ranged from 1.18 to 1.32. The transverse specimens had slightly better ductility and notched-strength ratios than the short-transverse specimens; their average strengths were almost undistinguishable.

### Charpy Tests

Charpy tests were made on standard V-notch specimens with an impact testing machine meeting the requirements of ASTM E23-64. Specimens broken at room temperature and at  $-65 \pm 3$  F gave the values listed in Table 23.

TABLE 23. CHARPY V-NOTCH VALUES OF 18Ni AND 9Ni-4Co STEELS(a)

		Average Charpy	Value, ft-lb	
	At Room To	emperature	At -	65 F
Steel and Condition	Longitudinal	Short Transverse	Longitudinal	Short Transverse
18Ni(300) VAR	12.2 ±2.0	13.3 ±4.4	13.9 ±2.0	12.4 ±1.7
18Ni(250) Air melt	16.1 ±3.0	14.5 ±1.4	14.1 ±1.5	14.7 ±0.1
18Ni(250) VAR	21.4 ±5.8	18.5 ±3.3	19.6 ±2.4	18.5 ±1.9
9Ni-4Co(250) VAR	26.1 ±1.7	24.6 ±4.6	26.0 ±1.9	22.8 ±2.0

<sup>(</sup>a) Averages are based on testing four specimens; the uncertainty of the observed averages is expressed by the standard deviation.

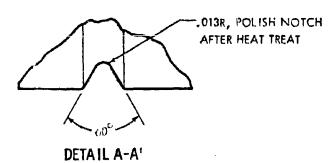
The differences in breaking energy between samples oriented in the longitudinal and in the short-transverse direction are not considered significant. Decreasing the test temperature to -65 F did not have a statistically significant effect on the Charpy values of any of the steels. Thus, the data indicate that the ductile-brittle transition temperature of the materials is below -65 F in Charpy tests.

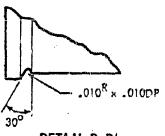
The Charpy values indicate that the energy required for ductile fracture was highest for the bainitic 9Ni-4Co steel and lowest for the 18Ni steel at the higher strength level. The "t" test for statistical significance of differences in averages indicates that the observed differences between the air-melted and vacuum-arc-remelted samples tested at the 250-ksi strength level is not attributable to experimental scatter.

#### Fatigue

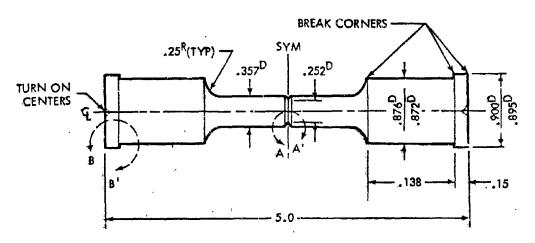
The high-cycle low-stress notched fatigue specimens, shown in Figure 16, were loaded axially in a BLH Model SF-10-U machine. The device is equipped with a preload

# A. HIGH CYCLE-LOW STRESS-COLLET ATTACHMENT





DETAIL B-B'



## B. LOW CYCLE-HIGH STRESS-THREADED ATTACHMENT

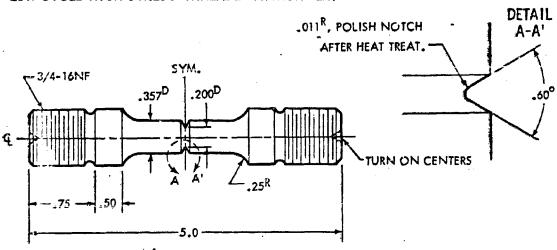


FIGURE 16. NOTCHED AXIAL FATIGUE SPECIMENS

 $K_t = 3$ .

maintainer. Attaching the specimens to the fixture with a collet device provided excellent axiality of load application. The high-cycle tests were conducted at 30 cycles/sec. The ratio of minimum load to the maximum load in the axial fatigue tests, or R factor, was 0.1. The stress concentration factor  $(K_t)$  was 3.

In general, the scatter in life values was small at high stress levels and normal near the endurance limit. The scatter from the trend line was more pronounced in tests on the 9Ni-4Co(250) steel than it was for the 18Ni steels. The endurance limits, at 10 million cycles, of the five steels shown in Figure 17 are:

•	E	Endurance Limit
Steel	Ksi	Percent of Ultimate Strength
D6AC(250)	92	
9Ni-4Co(250)	90	33
18Ni-Co-Mo(250) VAR	63	23
18Ni-Co-Mo(250) AM	60	22
18Ni-Co-Mo(300) AM	55	19

The chart shows that decreasing the tensile strength of vacuum-arc-remelted 18Ni(253) steel by 28,000 psi raised its endurance limit by 5000 psi. The air-melted steel performed as well as the vacuum-remelted steel in the fatigue tests.

The low-cycle high-stress fatigue tests were conducted in a modified ARC weld Creep Machine of 12,000-lb capacity. With this deadweight, lever-arm machine the elevator raised and lowered the loading weight cyclically until failure occurred. The R factor was 0.1 and the rate was 0.025 cycles/second. Figure 18 summarizes data obtained in the high-stress low-cycle fatigue evaluations.

Since the tests were run at stresses of 200 ksi and higher, failures occurred in less than 20,000 cycles. The maraging steel with the highest tensile strength had the shortest fatigue life at all stress levels investigated. Of the other materials, the 18Ni(250) vacuum-arc-remelted steel gave the best test performances. It and the other maraging steels seemed to fail from gradual growth of a crack originating in the notch. In the baintic 9Ni-4Co steel, on the other hand, failure typically occurred by growth or coalescence of numerous cracks that formed independently.

### Susceptibility to Stress-Corrosion Cracking

The susceptibility of the steels to stress-corrosion cracking was evaluated by subjecting them to a sustained axial tensile load under repeated cycles of immersion in NaCl solutions and drying. The loading was applied by deadweight machines; the specimen container was open to the atmosphere to allow the specimens to dry between immersions. The round, unnotched specimens shown in Figure 19 were degreased and cleaned in acetone immediately before starting a test. Two surface conditions, three chloride solutions, and three loading conditions were used for the studies.

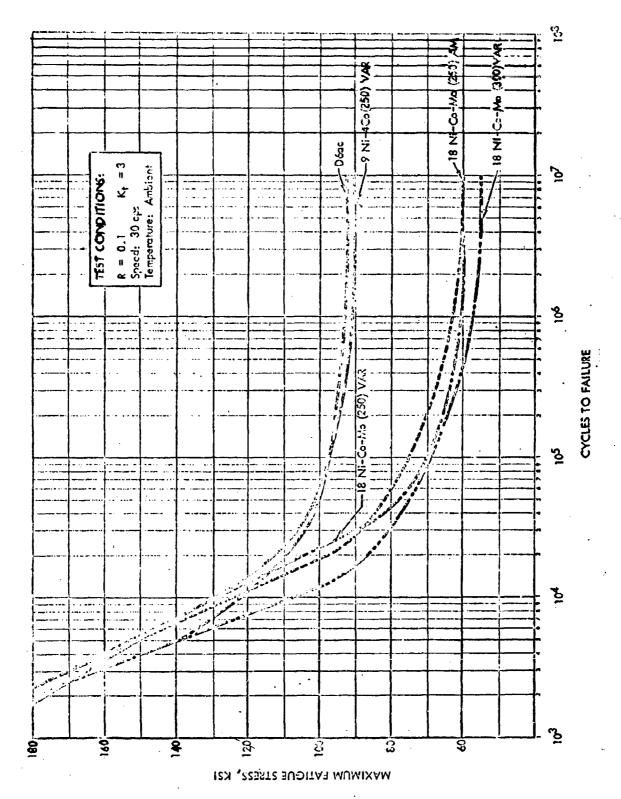
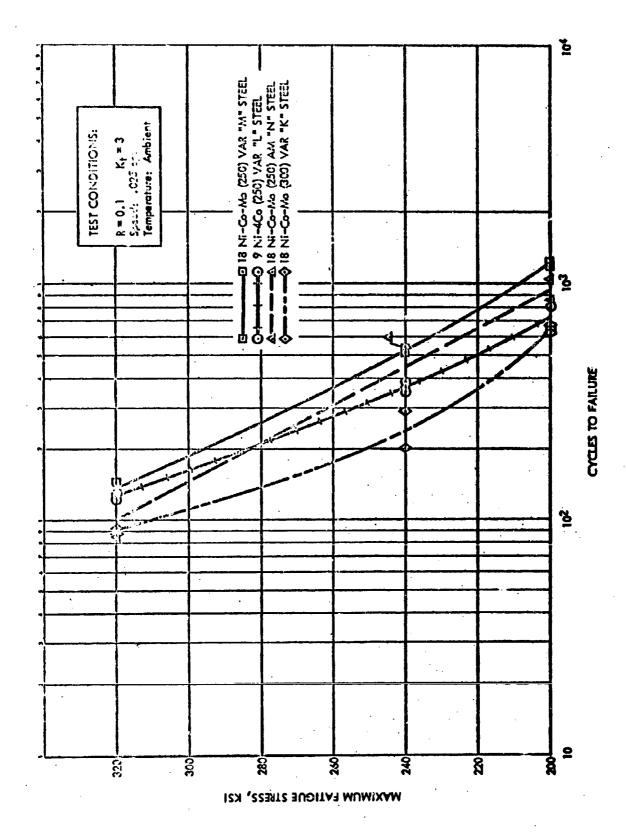


FIGURE 17. NOTCHED-AXIAL-FATIGUE PROPERTIES OF HIGH-STRENGTH STEEL FORGINGS



LOW-CYCLE - HIGH-STRESS NOTCHED-AXIAL-FATIGUE PROPERTIES OF HIGH-STRENGTH STEEL FORGINGS FIGURE 18.

Longitudinal Grain Direction.

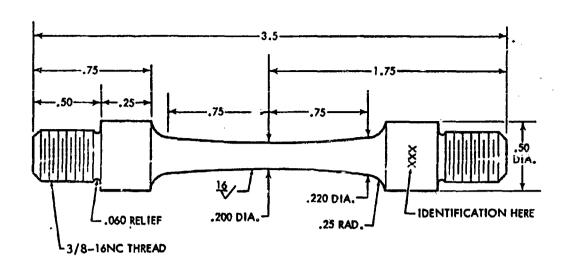


FIGURE 19. STRESS-CORROSION SPECIMEN

Table 24 lists results obtained on both air-melted and vacuum-arc-remelted 18Ni steels and on a vacuum arc remelted 9Ni-4Co(250) steel. The three failures, in 32 specimens, occurred in the radius of the specimen rather than in the minimum section. Examinations disclosed that the failures originated at the locations of severest end-grain condition. The tests showed that both the 18Ni(250) and the 18Ni(300) steels have good resistance to stress-corrosion cracking. It should be noted that both failures of the air-melted steel occurred at times in excess of the arbitrary 500 hours established as the normal test duration.

The general corrosive attack by the NaCl solutions on the surface of the 9Ni-4Co steel was more severe than it was on the maraging-steel specimens. Nevertheless, the tests indicate that the bainitic steel showed good resistance to stress-corrosion cracking. The two failed specimens exhibited large shear lips, and smaller cracks at the time of failure than the 18Ni samples.

### Fracture Toughness

The fracture toughness of specimens from the forgings was evaluated with the slowbend specimen shown in Figure 1 by the methods described previously in the section on "Forging Reduction Studies".

The data obtained in the slow-bend fracture-toughness tests are summarized in Table 25. The  $K_{nc}$  values are the most suitable for comparisons; the other values are included because they were used in the calculations.

In general, the data showed no particular forging to be better or worse than others in the same group. This means the toughness properties of the 18Ni(300) and the 9Ni-4Co(250) steels were not affected by heat-to-heat variations. The average  $K_{\rm nc}$  values and their standard deviations were:

Material	Longitudinal, ksi √in.	Short Transverse, ksi √in.
18Ni(300) VAR	51,9 ±1,24	54, 4 ±2.08
18Ni(250) VAR	$66.0 \pm 1.93$	66.3 ±2.07
18Ni(250) AM	57.7 ±1.24	58.3 ±3.2
9Ni-4Co(250) VAR	57.7 ±2.8	58.7 ±3.47

The average  $K_{nc}$  values were not very sensitive to specimen orientation with respect to grain flow. Presumably, this reflects the uniformity of deformation within the part chosen for study and the specimen locations. In all cases, the  $K_{nc}$  values were higher or better for the short-transverse than the longitudinal specimens. The scatter in individual measurements, however, was invariably larger among groups of short-transverse specimens. Only in the case of the 18Ni(300) steel, however, were the differences in means found to be statistically significant in the "t" test (at P = 0.05). The lengths by which the cracks increased in going from the proportional limit to the point of instability in the notched-bend tests were also greater for the short-transverse specimens. This suggests that they would withstand more crack propagation before failure.

The  $K_{nc}$  values for the 18Ni(250) air-melted steel were lower than those for its vacuum-arc-remelted counterpart. The difference is statistically significant. The  $K_{nc}$  values of the 9Ni-4Co steel are essentially the same as those for the air-melted maraging steel. Supplementary experiments indicated that the second heat treatment did not affect the fracture toughness of the bainitic steel.

TABLE 24. STRESS-CORROSION TEST DATA ON ROUND, UNNOTCHED TENSILE SPECIMENS WITH SHORT-TRAVERSE GRAINS EXPOSED

	Load,	Percent of		<u> Failures</u>		No Fai	iure
Conditions(a)	ksi	F <sub>tu</sub>	Specimen	Location(b)	Hour	Specimen	Fours
			18Ni(300) VAR F	orgings Code "K"			
В	260	90	<b>B</b> 7	тѕ	113	A7, A8	333
	260	90	<b>B</b> 8	TS	77	D7	335
C	218	75				D8, E7, E8	332
A	210	72.5	<b>A</b> 6	R.	456	<b>A</b> 5	528
••	210	72.5	<b>B</b> 6	TS	301	B5, D5, D6	50 <b>2</b>
	210	72.5 72.5	E5	R	350	E6	502 507
				orgings Code "N"			•
			TONICOOL VM P	orkinks code M			
В	242	90				A8, A7	411, 579
	242	90		<del></del>		B8, B7	406, 411
С	200	75	••			D7, D8	338
_	200	75	. ••		<b>~-</b>	E7, E8	338
A	200	75	••			A6, A5	521, 575
	200	75	<b>B</b> 6 ^	R	520	B5	545
	200	-75	D6	R	686	D5	504
	200	75				E6, E5	504, 819
		•	18 <u>Ni (250) VA</u> R F	orgings Code "M"		-	•
B	025			· · · · · · · · · · · · · · · · · · ·		40.45	. 440 ===
В	235 235	90 90				· A8, A7	446, 577
	<b>4</b> 30	<b>8</b> U				B8, B7	406, 446
C	195	75				D7, D8	333
-	195	75				D8, D7	300, 333
<b>A</b>	105	75				45 40	
A	195 1 <b>95</b>	75 75				A5, A6	503, 507
	195 195		ne	<b></b>	499	B5, B6	501, 504
	195 1 <b>9</b> 5	75 75	D6	R 	433	D5 E6, E5	504 501, 502
	-2-					w-, av	
		91	NI-4CO(250) VAR	Forgings Code "L'	-		
В	213	80	~ =			A8, A7	339, 575
	213	80	<b>B</b> 7	TS	191	B8	311
С	200	75				D7, D8	302, 338
_	200	75			·	E7, E8	338
A	200	75	<b>A</b> 5	re	\$50	AG	500
Λ	200	75 75	vo	. TS	570	A6 B5, B6	520 544
	200	75				D5, D6	504 503
	200	75		. =	-	E5, E6	503 502, 518

<sup>(</sup>a) A = Vapor-honed surface, 3-1/2% NaCl solution; cycle: 9 minutes wet, 51 minutes drying.

B = Vapor-honed surface, 3-1/2% NaCl solution; cycle; 9 minutes wet, 51 minutes drying; higher stress ratio than "A".

C = Hand-polished surface, 5% NaCl solution; cycle; 5 minutes wet, 15 minutes drying.

<sup>(</sup>b) TS = broke in test section; R = broke in radius.

TABLE 25. FRACTURE-TOUGHNESS PROPERTIES OF SPECIMENS TAKEN FROM EXPERIMENTAL FORGINGS(\*)

Grain Direction					į						
	Porging	Load, jest	Slope, Cra let/in.	Crack Depth, in, (b)	Load, kai	Siope, kd/in.	Crack Depth. in. (b)	Gnc, in1b/in. <sup>2</sup>	Knc,	.c. in1b/in. 2	Kc,
				18N	18NI (300) VAR Forgings Code "M"	rgings Code	. W.				
Longitudinal	₹	1.30	17.2	0.164	1.32	85.8	0.198	3	C:	13.6 5	ŝ
	<b>£</b>	1.20	74.1	6, 174	1.23	69.5	0, 187	2,58	) <b>4</b>	106.5	7 7
	Q	1.27	76.4	0,166	1.39	73.0	0, 177		70	183.3	3 2
	M	1.26	77.8	0, 163	1,36	73.3	0.176	84.6	50.4	116.4	55.7
Short transverse	<b>⋖</b>	1, 13	67.3	0.196	1.14	83.	0.207	7.76	S	1 801	Š
	m	1.20	71.5	0.184	1, 14	68.9	0, 197	95.2		114.4	3 %
	Q	1.27	72.2	0.182	1.40	68.7	0, 193	104.7	) ) )	7.071	3 8
	EL)	1.28	74.3	0.174	1.36	72.1	0, 183	96.7	54.0	119.2	57,2
				181	18NI (250) VAR Forgings Code "M"	erings Code	אַ				
Congitudinal	∢	1.80	80.8	0.152	1.86	76.4	0, 163	144.6	64.9	9 22	7.4
	M	1, 78	79.0	0, 157	2.00	76.1	0, 168	149.7	4.89	219.0	75.0
	Δ,	1.87	82.1	0.147	2, 17	78.8	0, 158	140.5	64.1	225.8	
	M	1. 83	79.3	0, 157	<b>3</b> .08	76.2	0, 167	159.2	68, 5	234.0	78.7
Short tramverse	∢	1,66	78.3	0, 199	1.8	74.7	0.176	145.7	4.58	0 866	6
	₽	1.75	78. €	0, 165	1.95	75.6	0, 174	162.7	. 68 8	225.4	
	Ω	1.73	79.0	0, 163	1.90	75.3	0, 174	148.6	66.0	215.7	75.6
	<b>L</b>	1.60	77.8	0, 167	1.78	71.1	0.187	141, 5	4.4	221.2	76.5
			-	187	18Ni (250) AM Pergings Code "N"	ging Code	ż				
Longitudinal	<	1.31	74.6	0.172	1.57	63.3	0.205	116.8	89	207.2	73.7
	m	 83	78.1	0,160	1,63	67.0	0.194	116,6	58.0	201.3	22.8
	Ω	1.62	80.6	0, 153	1.82	69.3	0.168	118.4	58.7	25.5	78.
	ш	-i 85	9.6	0.151	1.68	67.4	0.193	106.3	55,5	212.2	74.7
Short transverse	<b>«</b>	1.33	74.2	0.178	1. 58	62.3	0.812	111.3	57.5	220.6	<b>4</b>
	A	1.4	80.3	0, 192	1.73	69.8	0, 190	103.3	2.0	217.7	75.9
	Ω.	1.47	75,3	0, 174	1.72	65.0	0.80	133.7	0.83	244.8	80.5
	ш	1.4	76.2	0.174	1,68	65.9	0.201	121.1	29.50	28.5	2.5

TABLE 25. (Continued)

		<b>4</b>	Proportional Limit	Limit	1	Instability Point	sint			ļ	į
Grain Direction	Forging	Load, ksi	Slope, lai/in.	Crack Depth, in. (b)	Load, kai	Slope, kai/in.	Crack Depth, in. (b)	Gnc, in1b/in. <sup>2</sup>	Knc, kaí√in.	Gc, in1b/in. <sup>2</sup>	kai Çin.
	1			4- iN6	9Ni-4Co(250) VAR Forgings Code "L"	Forgings Co.	ie "L"				
	•	90	21.8	0.187	1.43	65.9	0,203	110.5	60.4	157.0	68.0
Longstudina	۲ ،	50.		177	7	66.3	0.202	108.5	89.9	182.4	73.3
	ma.	1.35	2.0	1 T *O	5 :	9 6		7 60	7. 1.	141.2	64.5
	Δ	1.35	73.6	0, 181	₹.	1.80	0. 18.	2.0			
•	म	1.18	71.9	0.186	1.36	9.99	0.201	91.7	55.1	139.8	į
										,	•
•	•	06.1	42.00	187	1.56	65.2	0.210	112.1	6.09	206.3	77.9
Short transverse	۱ ک	7.00	5 6	001	1 47	63.8	0.214	123.0	63.8	184.3	73.8
	22	1.28	1.60	0.188		2 5	198	87.7	53.9	147.9	0.99
	Ω	1.24	. s.	O. 173	T. 40	11.0	0. 190				
	m	1,29	78.2	0.174	1,45	71.6	0: 192	95.8		149.2	60.3

(a) Heat numbers given in Table 16; values are averages for three specimens.
(b) Calculated crack and notch depth at proportional limit or instability loads indicated. Specimen widths ranged from 0.4991 to 0.4996 for 18Ni steels and from 0.5008 to 0.5015 for 9Ni-4Co samples.

### EVALUATION OF INGOTS, SLABS, AND 4-INCH PLATE

The effects of ingot size on the properties of 18Ni(250) steel were investigated by Curtiss-Wright Corporation on material provided by Republic Steel Corporation. Tensile and fracture-toughness tests were made on specimens taken from ingots, forged slabs, and plates rolled to 4 inches.

Table 26 shows the ranges in chemical compositions found by analyzing samples taken from 18 locations in each of four ingots. The vacuum-arc-remelted ingots were produced from the air-melted and vacuum-degassed Heat No. 331290. Although the spread in composition tended to be a little wider in larger ingots, the differences are not significant.

### Tensile and Fracture-Toughness Tests

Table 26 also gives the ranges in mechanical properties, as the average of triplicate tests, determined on specimens taken from 18 locations in the three vacuum-remelted ingots. The reproducibility was poor, presumably because of the large size of the as-cast grains. Consequently, the data do not permit one to judge whether the properties varied with ingot diameter in the range from 18 inches to 32 inches. The ranges were wider, however, for values representing the largest ingot.

Stock from each ingot was forged to slabs 10 inches thick and 40 inches wide. Ultrasonic inspection showed the slabs to be sound. Mechanical-property data for specimens taken at slab locations representing different positions in the ingot are summarized in Table 27. They show that no particular location in the ingot was associated with unusually high or low property values. Specimen orientation did not affect the strength, ductility, or fracture-toughness values consistently. Among specimens taken from comparable positions, however, longitudinal bars usually had the highest yield strengths and transverse samples the best fracture-toughness values. Specimens taken in the short-transverse direction ordinarily gave the lowest yield strength, fracture toughness, and reduction in area values.

Specimens from slabs forged from the 32-inch ingot had better fracture-toughness values than those from similar locations in the 24-inch and 18-inch ingots. Since the differences were not so consistent between the 24-inch and 18-inch ingots, something more than ingot size may have been involved. The yield strengths were lowest for slabs from the 32-inch ingot and highest for those from the 18-inch ingot. This indicates, as would be expected, an inverse relationship between yield strength and planestrain fracture-toughness values.

Slabs from the two larger ingots were not rolled to plate 4 inches x 40 inches x 100 inches. One 10-inch-thick slab from each was rolled parallel to the longitudinal direction of the ingot (straightaway). The total reductions are equivalent to approximately 7 and 5 to 1 for the 32- and 24-inch ingots, respectively. A second slab from the 32-inch ingot (Heat No. 3960882) was rolled in a direction transverse to the original length of the ingot. Thus, the percentage reduction in cross rolling this plate was a little larger than the original reduction in the other direction in rolling the slab. Table 28 summarizes the properties of the plate.

TABLE 26. RANGES IN CHEMICAL COMPOSITIONS AND MECHANICAL PROPERTIES OF 18NI(250) INGOTS(2)

Heat Number:	331290	39608 <b>8</b> 2	3920872	3930871
Melting Practice: Ingot Size:	AM-D 43-Inch Octagon	VAR 32-Inch Round	VAR 24-Inch Round	VAR 18-Inch Round
С	0.016/0.024 <sup>(b)</sup>	0.017/0.026	0.017/0.025	0.015/0.023
Mn	0,11	0.07/0.09	0.07/0.09	0.09
P	0.003	0.004/0.005	0.005/0.C07	0.004/0.007
s	0.003/0.006	0.003/0.004	0.003	0.003/0.004
Si	0.06/0.07	0.09	0.07/0.09	0.07
Ni	17.70/17.90	17.78/17.90	17.78/17.95	17.80/18.00
Mo	4.87/5.02	4.88/4.98	4.86/5.00	4.95/5.05
Al		0.09/0.12	0.10/0.13	
Co	7.75/7.80	7.70/7.80	7.74/7.85	7.90
Ti	0.45/0.48	0.45/0.49	0.46/0.50	0.48/0.50
· <b>B</b>	0.0023/0.0025	0.0024/0.0026	0.0023/0.0026	0.0020/0.0035
Zr	0.021/0.025	0.020/0.027	0.022/0.027	0.023/0.025
Brinell Hardness	245/290	280/302	280/322	268/302
Ultimate Strength, ksi		188.0/250.5	210.5/246.0	218.3/248.5
0.2% Offset Yield Strength, 1/si		207.6/239.0	211.0/238.4	218.3/233.0
Elongation, %	_	1.0/10.2	1.0/8.8	1.0/5.7
Reduction in Area,	%	0.8/34.4	1.1/29.5	1.2/34.9
G <sub>Ic</sub> , in, -lb/in. <sup>2</sup>	** <del>**</del>	184/511	57/832	342/537

<sup>(</sup>a) The chemical analyses are ranges for determinations made at 18 locations in the ingots. The mechanical-property values are ranges for triplicate tests on longitudinal, transverse, and short-transverse specimens from the edge, middle, and center of slices taken from top and bottom ingot locations. The specimens were solution annealed 1 hour at 1500 F, air cooled, and aged 3 hours at 900 F.

The  $G_{Ic}$  values were determined on 0.400-inch-wide, 0.400-inch-deep bars with machined notches 0.060 inch deep; the bending width was 3.2 inches. The depths of the fatigue cracks ranged around 0.046 inch.

<sup>(</sup>b) Chemical composition in percent.

TABLE 27. TENSILE AND FRACTURE-TOUGHNESS VALUES FOR SPECIMENS TAKEN FROM 10 X 40-INCH SLABS OF 18Ni(250) STEEL(2)

	Т	ensile Propertie	6		Fracture
	Ultimate	0.2 Percent			Toughness
	Tensile	Offset Yield		Reduction	(Average
Specimen	Strength,	Strength,	Elongation,	in Area,	$G_{Ic}$ ),
Orientation	ksi	ksi	percent	percent	in1b/in.2
	Heat 1	No. 3930871, 18	-Inch Ingot, Top	Edge	•
T	254	242	8.3	28.5	330
ST	258	245.5	6,8	24.6	236
L	257	245	8.2	29.0	274
	Heat 1	No. 3920872, 24-	Inch Ingot, Cop	Edge	
T	252.6	241.7	4.0	18.9	215
ST	253.4	239.8	6.5	29.9	237
L	247.6	235.9	7.3	32.5	362
	Heat 1	No. 3960882, 32-	Inch Ingot, Top	Edge	
T	253.6	241.7	5. 5	20.9	<del>4</del> 56
ST	256.3	241.5	6.3	21,0	372
L	253.8	240.5	5,8	19.6	390
	Heat 1	No. 3930871, 18-	-Inch Ingot, Top	Middle	
Т	258	246.0	6.0	26.8	355
ST	260	249.0	4.0	13.8	247
L	260	-249.0	7.0	23.7	340
	Heat 1	No. 3920872, 24	-Inch Ingot, Top	Middle	
T	253	239.5	7.0	33.6	299
ST	254.4	238.8	5.5	15,8	259
L	253.8	239.4	7.0	31.6	280
	Heat !	No. 3960882, 32	-Inch Ingot, Top	Middle	
T	254.1	242.4	5.8	24.6	394
ST	254.3	243.5	5.5	18,1	280
L	253,0	240.2	5.5	18.0	432
	Heat 1	No. 3930871, 18	-Inch Ingot, Top	Center	
<b>T</b>	257	249	9.3	32.8	384
ST	258	246	6.5	22.2	246
L	258	253	5.5	24, 1	322

TABLE 27. (Continued)

		Tensile	Properties		Fractu.
	Ultimate	0.2 Percent			Toughne
	Tensile	Offset Yield		Reduction	(Avera
Specimen	Strength,	Strength,	Elongation,	in Area,	$G_{Ic}$ ),
Orientation	ksi	ksi	percent	percent	inlb/in
	Heat I	No. 3920872, 24	-Inch Ingot, Top	Center	
T	251.4	237.4	5.5	28.1	318
ST	251.1	238.3	5.0	18.7	232
L	252,0	239.5	6.0	30.7	412
	Heat I	No. 3960882, 32	-Inch Ingot, Top	Center	
T	249.7	233.8	6.8	25.7	287
ST	250.5	235.3	4.7	17.2	252
L	2 <b>4</b> 2.3	235,4	7.5	34,1	308
	Heat 1	No. 3930871, 18	-Inch Ingot, Bot	tom Edge	
T	260	248.4	8.7	32.6	238
ST	257	245.0	7.8	27.8	258
L	259. <b>4</b>	248.4	7.0	26.8	230
	Heat J	No. 3920872, 24	-Inch Ingot, Bot	tom Edge	
T	259.5	248.9	, <b>7.</b> 8	31.2	299
ST	259.7	248.1	7.0	32.6	349
L	259.5	250.2	7.5	30.5	267
	Heat 1	No. 3960882, 32	2-Inch Ingot, Bot	tom Edge	
T	256.2	245.7	7.0	31.6	361
ST	255.8	245.4	. 3.8	9.8	272
L	256.4	245.1	8.5	37.5	3 <b>54</b>
	Heat 1	No. 3930871, 18	B-Inch Ingot, Bot	tom Middle	
T	260	250.9	7.5	32.0	339
ST	259.5	252.7	5.5	21.7	249
L .	262.5	253.3	5.5	18	248
•	Heat	No. 3920872, 24	4-Inch Ingot, Bot	tom Middle	•
T	255.2	238.5	7.5	32.9	277
ST	254.2	238.6	6.0	13.1	219
L	255.8	241.8	6.5	32.1	277

TABLE 27. (Continued)

		Tensile :	Properties		Fracture
Specimen Orientation	Ultimate Tensile Strength, ksi	0.2 Percent Offset Yield Strength, ksi	Elongation, percent	Reduction in Area, percent	Toughness (Average G <sub>Ic</sub> ), inlb/in. 2
•	Heat 1	No. 3960882, 32	-Inch Ingot, Bott	om Middle	
Т	250, 1	238.1	5.0	19.2	309
ST	234, 1	243.5	3.7	7.6	264
L	256. <b>4</b>	248.6	5.0	24.8	297
	Heat !	No. 3930871, 18	-Inch Ingot, Bott	tom Center	
T	260,8	250.7	7.8	33.1	338
ST	259.9	249.5	6.3	16.7	327
L	262.1	251.3	5.3	16.5	286
	Heat !	No. 3920872, 24	-inch Ingot, Bott	tom Center	
. <b>T</b>	251.2	238,1	8.0	29.3	245
ST	249.9	237.9	2,5	8.0	195
L	252.0	238.7	6.5	29.5	333
	Heat	No. 3960882, 32	-Inch Ingot, Bot	tom Center	
T	253,3	245.8	5.8	21,1	317
T	252.2	244.8	6.5	14.4	271
L	255.6	246.9	6.5	21.2	381

<sup>(</sup>a) Each value is an average for several observations, usually for three specimens. The specimens were solution-annealed 1 hour at 15.0 F, air cooled, and aged 3 hours at 900 F.

TABLE 28. TENSILE AND FRACTURE-TOUGHNESS PLATE VALUES FOR SAMPLES TAKEN FROM 4-INCH PLATES OF 18Ni(250) STEEL(a)

		Tensile 1	Properties		Fracture
	Ultimate	0.2 Percent			Toughness
	Tensile	Offset Yield		Reduction	(Average
Specimen	Strength,	Strength,	Elongation,	in Area,	G <sub>Ic</sub> ),
Orientation	ksi	ksi	percent	percent	in, -lb/in.
	Heat	No. 3920872, 24	Inch Ingot, Top	Edge	
т	266.5	257.1	4.0	21.8	178
ST	265.9	257.1	6.0	17.8	177
L	270.3	264.8	6.0	32,3	193
	Heat	No. 3960882, 32	-Inch Ingot, Top	Edge	
Т	259.6	245.9	8.2	28.7	159
ST	257.7	244.5	5.3	17.4	164
L	260.5	247.3	6.8	31.0	178
	Heat	No. 3960882T, 3	2-Inch Ingot, To	op Edge	
<b>T</b> .	276.6	264.9	6.8	26.3	219
ST	239.0	260.9	2.0	2.8	214
L	277.1	265.4	5.5	19.8	202
	Heat	No. 3920872, 24	-Inch Ingot, Top	Middle	
Т	267,5	260.2	2.8(b)	18.8	194
ST	265.6	255.9	2.5	16.2	183
L	269.9	262.5	3.3	22.7	154
	Heat	No. 3960882, 32	-Inch Ingot, Top	Middle	
т	255, 1	244.4	7.5	28.5	141
ST	254.3	243.9	3.0(b)	11.5	161
L	257.5	246.1	6.7	30.0	157
	Heat	No. 3960882T, 3	2-Inch Ingot, To	op Middle	
Т	255.9	244.5	7.3	29. 1	269
ST	254.9	243.9	1.5	2.8	212
L	257.2	246.5	9.0	30. <b>4</b>	194
-	Heat	No. 3920872, 24	-Inch Ingot, Top	Center	,
Т	265,8	257.6	3.3(b)	31.8	221
ST	268.1	259.6	3.5	15.1	164
L	266.7	259.8	4.0	28.6	206

TABLE 28. (Continued)

			Properties		Fracture
	Ultimate	0.2 Percent			Toughness
	Tensile	Offset Yield		Reduction	•
Specimen	Strength,	Strength,	Elongation,	in Area,	G <sub>Ic</sub> ),
Orientation	ksi	ksi	percent	percent	inlb/in.
	Heat	No. 3960882, 32	Inch Ingot, Top	Center	
${f T}$	256.3	248.0	6.5	26.9	166
ST	254,7	244.0	3. 0 <sup>(b)</sup>	8.0	134
L	257.1	242.8	8.0	19.7	177
	Heat	No. 3960882T, 3	2-Inch Ingot, To	p Center	
T	257,2	246.1	7.5	29.3	254
ST	254, 1	242.4	2.8	3.8	198
L	257.2	246.4	9.0	32.3	169
	Heat	No. 3920872, 24	Inch Ingot, Bott	tom Edge	
Т	267.0	258.3	6.3	26.2	192
ST	264.7	255.0	4,5(b)	19.5	149
L	268.3	259.9	2.5(b)	29.9	156
	Heat	No. 3960882, 32	Inch Ingot, Bot	tom Edge	
T	260.3	250.5	5,5(b)	27.1	166
ST	253.1	240.9	2.5(b)	22.5	168
L	261.6	250.8	7.8	33.1	138
	Heat	No. 3960882T, 3	2-Inch Ingot, Bo	ottom Edge	
т	260	244.5	7.5	2 <b>6.</b> 9	250
ST	259.1	240.9	5.0	13.1	253
L	270.6	255,2	7.5	30.0	211
	Heat	No. 3920872, 24	Inch Ingot, Bot	tom Middle	
Т	270.7	260,2	4.5	26.2	233
ST	267.2	256.9	4.3	10.1	162
L	270.6	259.9	4.0	29.5	203
	Heat	No. 3960882, 32	Inch Ingot, Bot	tom Middle	
T	264.2	258,7	5.8	23.1	165
ST	245.9	254, 1	2.3	4.3	156
L	262.7	255.7	7.5	35,5	153

TABLE 28. (Continued)

		Tensile 1	Properties		Fracture
Specimen Orientation	Ultimate Tensile Strength, ksi	0.2 Percent Offset Yield Strength, ksi	Elongation, percent	Reduction in Area, percent	Toughness (Average G <sub>Ic</sub> ), inlb/in. <sup>2</sup>
	Heat 1	No. 3960882T, 3	2-Inch Ingot, Bo	ttom Middle	
Т	258. 1	244.1	3,5	28.3	156
ST	248,5	243.9	1.0		192
L	261.3	245.3	6,3	29.6	184
	Heat :	No. 3920872, 24	-Inch Ingot Botto	m Center	
T	263.7	252.7	4.8	24, 1	172
ST	262,5	257,2	3,0	15, 1	164
L	264.5	256.5	2.3 <sup>(b)</sup>	30.5	194
	Heat :	No. 3960882, 32	Inch Ingot, Boil	tom Center	
T	262.3	258.4	4.0	30.4	146
ST	264.2	259.7	2.0	5.6	127
L	263.2	257.4	5.0	32.1	156
	Heat	No. 3960882T, 3	2-Inch Ingot, Bo	ttom Center	
T	258.0	251.0	5.3	29.7	188
ST	205,0	<b></b>	1.3	1.0	145
L	258.1	250,8	3,5	21,2	133

<sup>(</sup>i) Each value is an average for several observations, usually for three specimens. The specimens were solution annealed 1 hour at 1500 F, air cooled, and aged 3 hours at 900 F.

<sup>(</sup>b) Specimens broke outside the gage marks.

The 4-inch plates had higher yield strengths than specimens taken from slabs; the average difference amounted to about 12 ksi. Rolling had no consistent effect on tensile reduction in area values, but impaired the elongation and fracture-toughness values. The latter change was quite pronounced, amounting to a drop of almost half the level for specimens taken from the 10-inch slabs. Although the cause is unknown, other investigators have noticed that plane-strain fracture-toughness values for maraging steels drop precipitately as yield strengths increase over approximately 245 ksi. When the comparison is restricted to plate samples, however, the G<sub>IC</sub> values are relatively independent of yield strength. For instance, the average fracture-toughness value for plates with yield strengths exceeding 250 ksi did not differ significantly, statistically, from that of plates with yield strengths below 245 ksi.

The average fracture-toughness value of samples from top cuts were better ( $G_{IC}$  = 190) than those for specimens with the same orientation taken from bottom cuts ( $G_{IC}$  = 171), but the differences were neither completely consistent nor statistically significant. The tensile ductility and yield strengths were poorer for samples taken in the short-transverse direction. The fracture-toughness values for edge, middle, and center locations were essentially identical.

Rolling practice and ingot size apparently influence the mechanical properties of the heat-treated 18Ni(250) steel to some extent. Plate straightaway rolled from the 24-inch ingot gave more consistent data and slightly higher strengths than straightaway-rolled plate from the 32-inch ingot. On the other hand, the cross-rolled plate from the large ingot had the best fracture toughness. The plate rolled from the 32-inch ingot had the poorest  $G_{IC}$  values. This difference, attributed to rolling practice, is judged to be a real effect based on the Student's "t" test for statistical significance. The same type of statistical analysis indicates that the apparently superior fracture toughness of the product from the 24-inch ingot, compared to that of the straightaway-rolled 32-inch ingot product, is not due to experimental scatter. In this case, however, it is difficult to decide whether the variation in fracture toughness resulted from differences in rolling reduction alone or from ingot size.

### Metallographic Examinations

The inclusion contents of ingot, slab, and plate products from the three vacuum-arc-remelted heats of 18Ni(250) steel were considered uniformly high from top to bottom and from edge to center locations. On the JK system for classifying inclusions, all materials had the following ratings:

D, Fine; 4-5

D, Heavy; 2-3

Specimens from ingots developed light- and dark-etching areas. The white spots were caused by retained austenite, and they had lower hardnesses. The size and number of the austenitic spots were not related to location in the ingot or ingot size. The microstructure of specimens from slabs and plates were more homogeneous. The grain sizes were smaller than the characteristic size of air-melted ingots, and the differences in hardness between light- and dark-etching spots were smaller.

#### PROCESSING AND EVALUATION OF SHEET AND PLATE

The objective of this phase of the research program was to determine the optimum conditions for mill processing and heat treating 18Ni steel to strengths of 300-and 250-ksi, and 9Ni-4Co steel to 250- and 200-ksi strength levels. Studies were made on both sheet and plate rolled from steels produced by different melting and deoxidation practices. Tensile and fracture-toughness tests were used to judge the effects of the following processing variables:

- (1) Hot-rolling temperature for plate
- (2) Hot-rolling temperatures for sheet
- (3) Cross rolling
- (4) Thickness of the product
- (5) Solution-treating temperature
- (6) Aging temperature.

Stress-corrosion tests were run on some products. A variety of fracture-toughness specimens were employed depending on the gage of the product being evaluated.

## Processing Studies on 18Ni Sheet and Plate

### Melting and Fabrication Background

The 250-ksi grade of this steel was produced in ingot form by the Republic Steel Corporation and the U.S. Steel Corporation. The Vanadium Alloys Steel Company produced the 300-ksi grade. Table 29 indicates the initial heat numbers involved and the final heat numbers assigned by Republic when vacuum-arc remelting (VAR) was used during processing. These ingots were converted into forged shapes and then rolled to the flat-rolled products indicated. The studies were carried out by Republic Steel Corporation.

TABLE 29. BACKGROUND INFORMATION ON 18NI MARAGING-STEEL HEATS USED FOR ROLLING AND HEAT-TREATMENT STUDIES

Type Steel:		18Ni (250 ksi)	·	18Ni (300 ksi)
Original Producer:	U.S. Steel	Repu	iblic	Vanadium
Producers' Heat Number:	X53088	32212	90 AM	08749
Ingot Remelter:	Not remelted	Not remelted	Republic	
Final Heat Number Assigned:	X53088	3321290	3930864	08749
Melting/Deoxidation Practice:	AM	AM-D	VAR	VAR
Republic Specification:	RSC-AF-1 CI I	RSC-AF-1 Cl I	RSC-AF-1 Cl II	RSC-AF-1 CHIL
Thickness of Plate Product Produced, inch:	0.375 0.750 3.0	0.375 0.750 3.0	0.375 0.750 3.0	•••
Gage of Sheet Products Produced, inch:	0.045 0.160	0.045 0.160	0.045 0.160	0.045 0.160
Mill Products Destination:	Aerojet -General	Aerojet-General	Aerojet-General	Aerojet -General

### Testing Conditions

For this part of the evaluation of the 18Ni maraging steel, the sheet and plate specimens were solution annealed for 1 hour at 1500 F and air cooled. Then they were aged 3 hours at 900 F, air cooled, and tested.

The starting material for the plate-rolling studies was in the form of  $4 \times 16$ -inch forged section. The sheet-processing studies were made on 0.4-inch-thick sheet bar processed from the  $4 \times 16$ -inch sections.

Tensile tells were made on standard strip specimens.

Two kinds of fracture-t ghiess specimens were employed:

- (1) For the 3/8-inc plate, precrs at 0.02 to 0.04-inch-deep cracks) nonstandard Charpy bars, 0.00 x (394 inch
- (2) For the 0.16-inch sheet, 3 with the center-notched tensile specimens of the type shown in Figure 20.

Comparisons among the precracked Charpy specimens were by ed on W/A values (in.-lb absorbed/in, <sup>2</sup> of area under the notch). The fracture toughness of the center-notch tensile specimens was judged on the basis of their N/YS ratios. That is, the ratio of the fracture load divided by the original uncracked area of the no ched specimen to the 0.2 percent offset yield strength.

# Hot-Rolling Studies - 3/8-Inch Plate

Figure 21 shows that varying the hot-rolling temperature from 1950 to 2150 F had no significant effect on the tensile propert as of 18Ni (250) vacuum-arc-remelted steel. Tests on the comparable air-melted steel (Heat No. 3321290), which was also the parent heat for the VAR steel, gave similar results, except that the dip in yield strength associated with rolling at 2050 F was more pronounced, being about 17 ksi. Since the effect was noted in studies on both materials, it appears to be real.

From the standpoint of fracture toughness, 1950 F is the best rolling temperature. It developed the following combination of properties:

Yield strength, ksi 284 for air melted, 278 for VAR

W/A, in.-1b/in.<sup>2</sup> 675 for air melted, 1240 for VAR

Thus, the test data also indicated that vacuum-arc remelting improves the fracture toughness of maraging steel.

## Hot Rolling Studies - 0, 160-Inch Sheet

The sheet-rolling studies were made on three heats of 18Ni steel. The effects of nine combinations of sheet-bar and sheet-rolling temperatures on mechanical properties of the maraging steel are summarized in Table 30. The table shows that the (300) VAR material from Heat No. 08749 was more sensitive to variations in rolling temperature

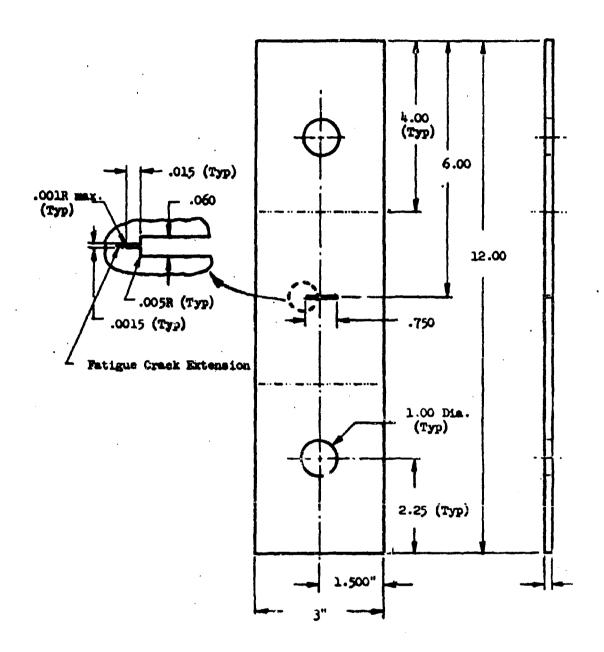


FIGURE 20. CENTEK-' OTCH TENSILE SPECIMEN FOR EVALUATING THE 0.045 ANL 0.160-INCH-THICK SHEET MATERIAL

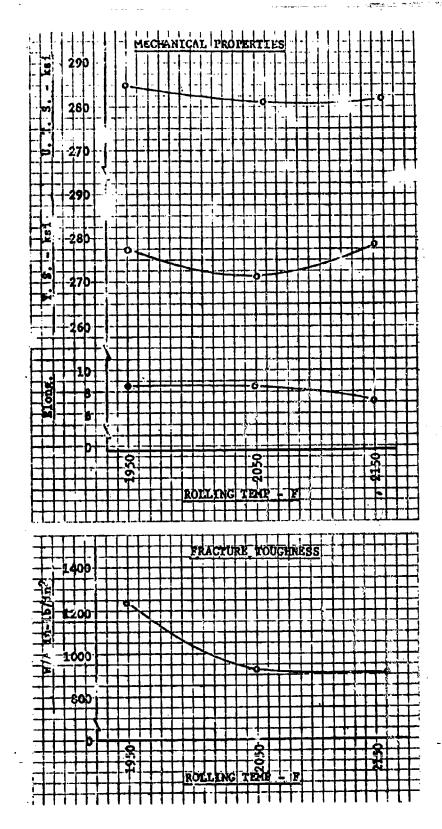


FIGURE 21. THE EFFECT OF ROLLING TEMPERATURE ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 18 Ni(250) VAR, HEAT 3920864

TABLE 30. EFFECT OF ROLLING TEMPERATURE ON PROPERTIES OF 0.160-INCH-THICK SHEET OF 18Ni STEEL

Code Number	Rolling Temperature, F	Tensile Strength, ksi	0.2 Percent Yield Strength, ksi	Fracture Toughness N/YS#
	Shee	t Bar Rolled :	it 1950 F	
(300) VAR	1450	282.5	275.5	0. 635
Ht. 08749	1550	287.5	282.0	0, 690
	1650	<b>,279.</b> 0	271.0	0,700
(250) VAR	1450	284.0	279.0	
Ht. 3920864	1550	279.5	273.0	0.680
	1650	276.0	272. 5	0.530
(250) AM-D	1450	286.0	283.0	0. 525
Ht. 3321290	1550	288.0	282.0	0,460
	1650	285.0	278,0	0.500
	Shee	t Bar Rolled	at 2050 F	
(300) VAR	1450	296, 5	296. 5	0,440
Ht. 08749	1550	282.0	276.0	0.700
, 0011,	1650	288.C	283.5	0, 620
(250) VAR	1450	285.0	281.0	0. 560
Ht, 3920864	1550	281.0	273, 0	0,510
	1650	274.5	267. 5	0,550
(250) AM-D	1450	284.0	278.0	0, 510
Ht. 3321290	1550	- 282.0	275, 5	0.525
	1650	274.0	266. 5	0.610
•	Shee	t Bar Rolled	at 2150 F	
(300) VAR	1450	295.0	292.0	0.500
Ht. 08749	1550	280,0	276.0	0.660
	1650	284.0	278.0	0, 650
(250) VAR	1450	279.5	274.5	0, 420
Ht. 3920864	15! 0	284, 5	280.0	0.530
<b>.</b>	1650	283.0	276.0	0.610
(250) AM-D	1450	281.0	276.9	0.550
Ht. 3321290	1550	285.0	274.0	0,550
	1650	278,0	270.0	0.490

Two linal strength of center notched sensile spectrage divided by the yield strength.

than the other steels. The highest strength resulted from rolling sheet at 1450 F after plate rolling at 2050 or 2150 F, but sheet bar that had been rolled at 1950 F gave more consistent results. The fracture toughness of the 18Ni (300) VAR material appeared to be superior to that of the other two materials. In general, however, the 18Ni (250) VAR sheet material did not exhibit better fracture toughness than its air-melted counterpart.

Based on these test results, it was concluded that the following rolling temperatures resulted in the best combination of strength and toughness in 18Ni steel:

Plate and sheet bar rolling

1950 F

Sheet rolling

1550 F .

# Cross-Rolling Studies

The cross-rolling studies were made on stock from Heat No. 3920864 which had been vacuum-arc remelted.

For the plate studies, a  $2-1/4 \times 12$ -inch section was rolled to 3/8-inch plate at 1950 F with 0, 50, and 67 percent transverse reduction (with respect to the original rolling direction). Figure 22 shows that the anisotropy reflected by tensile properties was decreased by the 50 percent cross-rolling treatment. In the plate which received 67 percent reduction by transverse rolling, anisotropy reappeared; the transverse specimens having higher strengths.

The fracture-toughness data in Figure 22 indicate that cross rolling was beneficial. This is somewhat surprising because the W/A values of the precracked Charpy bars seem to be independent of specimen orientation.

## Cross Rolling

The effects of cross rolling in reduction schedules in reducing sheet bar to sheet were also investigated. The 0.64-inch-thick sheet bar had been rolled at 1950 F, with 67 percent cross rolling from a 4-inch section. That stock was then rolled at 1550 F to 0.16-inch-thick sheet, with various additional cross rolling.

Figure 23 shows the properties of sheet produced by the three rolling schedules. Cross rolling had no significant effect on tensile strengths. This agrees with common experience. The chart also indicates that 50 percent reduction by cross rolling lowered, and 67 percent reduction improved, the fracture toughness. The changes are difficult to account for because N/YS values are quite similar for specimens taken parallel and transverse to the original rolling direction. Perhaps the apparent differences were caused by some factor other than the intended variable (cross rolling).

#### Heat-Treatment Studies

It had been reported that re-solution annealing of mill-annealed maraging steel plate, prior to aging, resulted in a variation in tensile and fracture-toughness properties. Since reproducibility was considered important, this point was investigated.

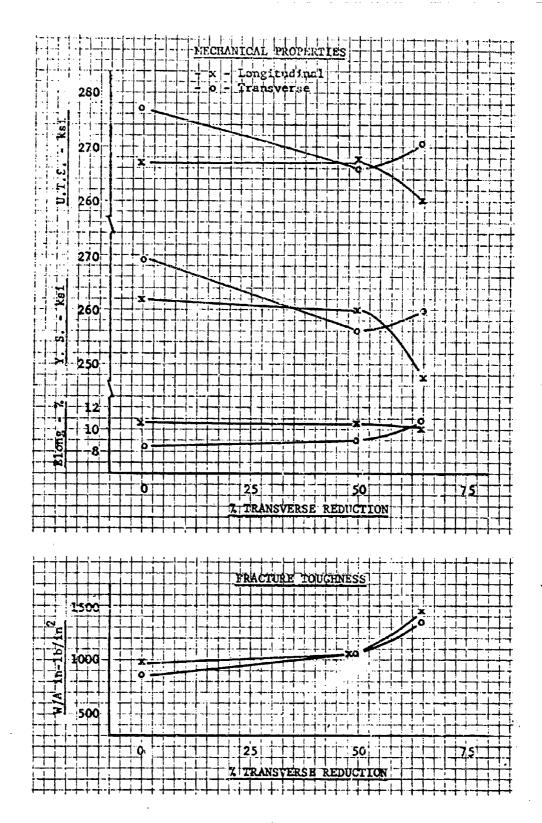


FIGURE 22. THE EFFECT OF CROSS ROLLING ON THE MECHANICAL A.(D FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 18Ni(250) VAR, HEAT 3920864

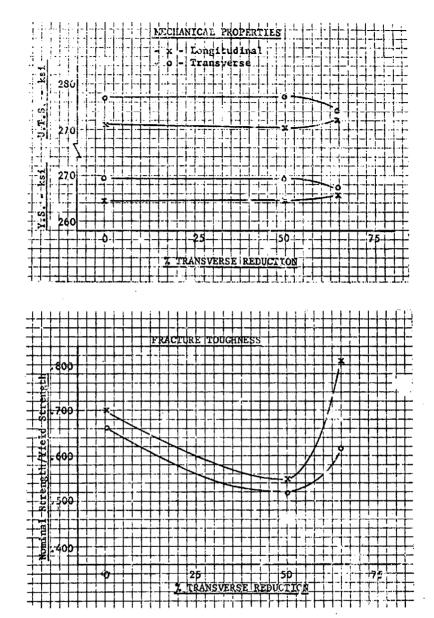


FIGURE 23. THE EFFECT OF CROSS ROLLING C . THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 160-INCH SHEET IN HEAT-TREATED 18Ni(250) VAR, HEAT 10864

Sheet and plate samples were heat treated in a variety of ways:

- (1) Specimens were subjected to two 1-hour solution-annealing treatments at 1500 F, 1550 F, 1600 F, and 1650 F. Comparison samples were given a single treatment at the same temperatures. All of these samples were aged at 900 F for 3 hours.
- (2) Samples solution treated for 1 hour at the temperatures mentioned above were aged at 950 F and their properties compared with samples aged at 900 F.

Solution Annealing. Figure 24 shows that 3/8-inch plates solution annealed at 1500 F developed higher strengths than those heat treated at the other temperatures investigated. The second annealing treatment at that temperature or at 1550 F, however, lowered the strengths slightly. The effect of doubling the time appeared to be roughly equivalent to raising the solution-annealing temperature b; 50 F. Tensile elongation values were not affected by variations in solution-annealing schedules.

The chart also shows that the re-solution annealing at 1500 F impaired the fracture toughness of sheet previously annealed at that temperature. On the other hand, the double treatments a 1600 or 1650 F improved fracture toughness without impairing yield or ultimate strength.

Figure 25, which summarizes the data for 0.160-inch sheet, shows that the tensile properties developed by a single-solution anneal were independent of the annealing temperature. This was not true for the plates from the same heat. Repeating the solution treatment at 1500 F raised the strength; repeating the solution anneal at any of the three higher temperatures lowered the strength about 5000 psi.

Repeating the solution-almealing treatment at the same temperature had no effect on the fracture toughness of the sheet in the aged condition. This lack of response to the second solution treatment did not agree with the results on the plate specimens. Solution annealing at 1500 F or 1650 F developed better fracture toughness than annealing at intermedial, temperatures.

Aging. The response of solution-annealed maraging steels to aging at 850 F to 950 F is a nonlinear function of the time at temperature. The sensitivity to differences in aging time seems to be more pronounced at lower aging temperatures. Because it is difficult to control temperatures closer than  $\pm 25$  F in commercial operations, an aging temperature of 925  $\pm 25$  F seems most likely to give consistent results. Therefore, experiments were conducted in order to determine the variations in mechanical properties to be expected among sheets and plates aged at 900 F and 950 F.

Figure 26 shows that plates solution treated at 1600 F or 1650 F developed equivalent mechanical properties after aging at either 900 or 950 F for 3 hours. In samples solution treated at 1500 or 1550 F, aging at 900 F developed better strength and toughness than aging at 950 F. The best combination of properties was developed by solution annealing at 1500 F and aging at 900 F.

In general, the tensile properties of sheet samples showed the same responses to variations in solution and aging treatments as the plate samples. Figure 27 shows

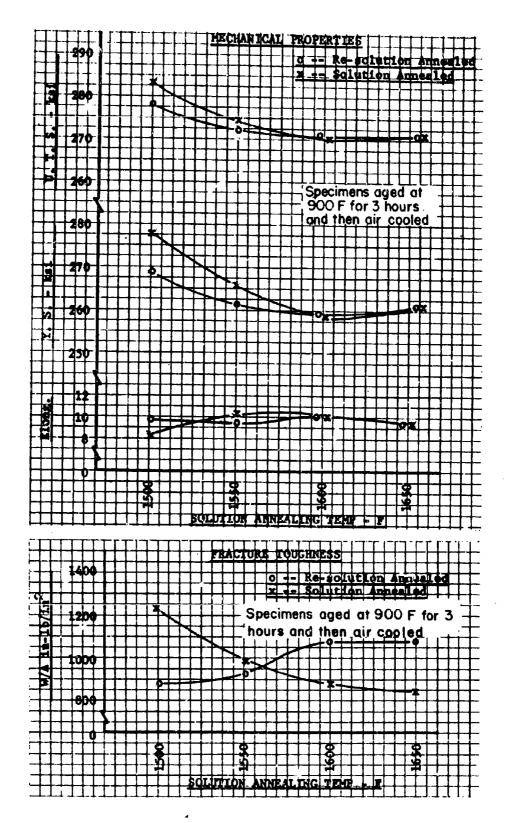
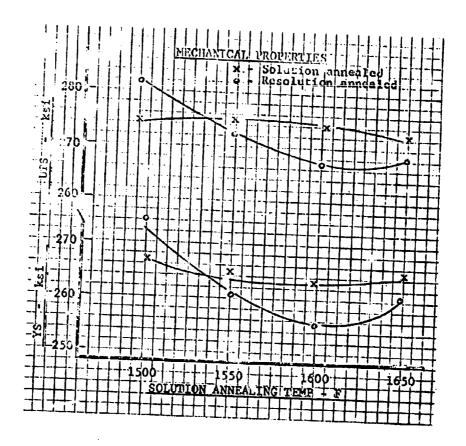


FIGURE 24. THE EFFECT OF SOLUTION-ANNEALING TEMPERATURE AND RE-SOLUTION ANNEALING ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH-PLATE IN HEAT-TREATED 18Ni(250)VAR, HEAT 3920864



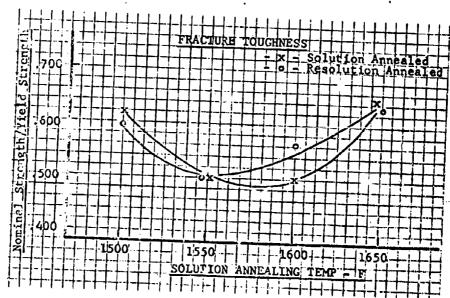


FIGURE 25. THE EFFECT OF SOLUTION-ANNEALING TEMPERATURE AND RE-SOLUTION ANNEALING ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 0. 160-INCH SHEET IN HEAT-TREATED 18Ni(250) VAR, HEAT 3920864

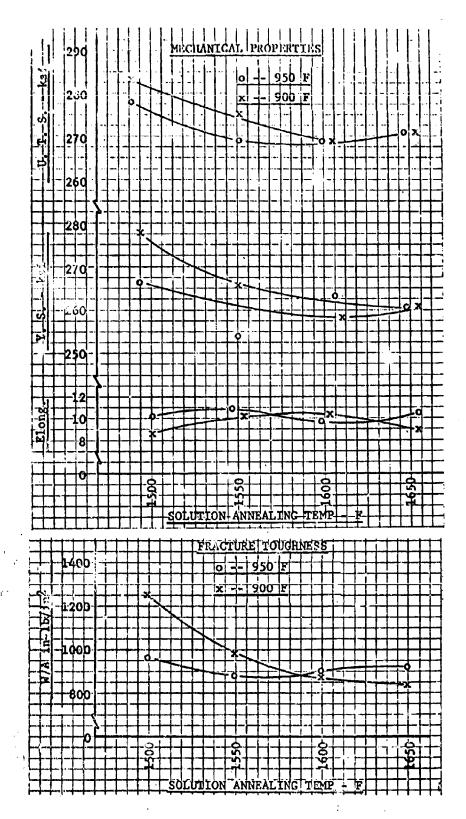
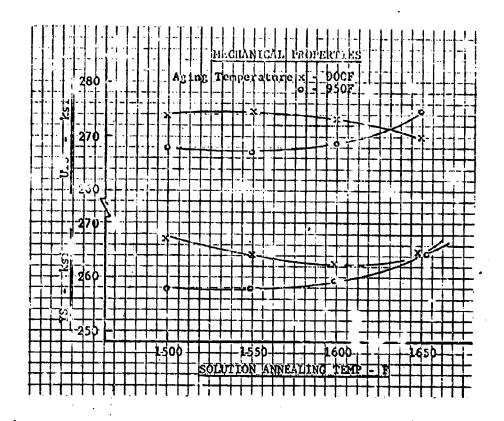


FIGURE 26. THE EFFECT OF SOLUTION ANNEALING AND AGING TEMPERATURES ON THE MECHANICAL AND FRACTURE- LOUGHNESS PROLERTIES OF 3/8-1NCH PLATE IN HEAT-TREATED 18Ni(250) VAR, HEAT 3920864



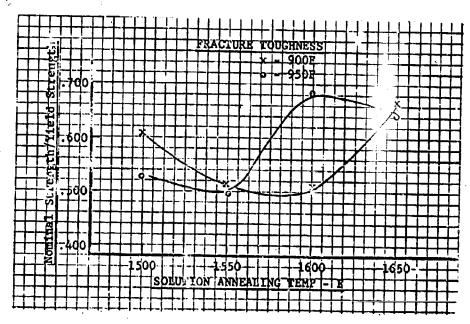


FIGURE 27. THE EFFECT OF SOLUTION ANNEALING AND AGING TEMPERATURES ON THE MECHANICAL AND FRACTURE TOUGHNESS PROPERTIES OF 0, 160-INCH SHEET IN HEAT-TREATED 18Ni(250) VAR, HEAT 3920864

that, among samples solution treated at 1500 F or 1550 F, aging at 900 F developed strengths about 6-ksi higher than aging at 950 F. On the other hand, samples solution annealed at 1600 F or 1650 F were relatively insensitive to differences in aging temperature. The strengths of sheet and plate samples were approximately the same when intermediate annealing temperatures were employed.

Fracture-toughness values for the sheet specimens showed significant, although unexplained, differ inces between aging treatments. The best combination of properties seemed to result from solution annealing at 1650 F. Such specimens exhibited equivalent and good toughness values after aging at either 900 F or 950 F. So far as consistency is concerned, this agrees with the properties for plates.

The heat-treating studies indicate that the most consistent properties in 18Ni maraging steel will be obtained by solution annealing at 1650 F and aging at  $925 \pm 25$  F.

# Processing Studies of 9Ni-4Co Sheet and Plate

## Melting and Fabrication History

This grade of steel was produced in ingot form by three steel companies. Table 31 indicates the heat numbers and prior history of the steels. These processing studies on flat-rolled products were conducted by Republic Steel Corporation.

TABLE 31. BACKGROUND INFORMATION ON 9Ni-4Co ALLOY STEELS USED FOR ROLLING AND HEAT-TREATMENT STUDIES

Original Producer and Heat Number	Final Heat Number	Melting/ Deoxidation Practice	Republic Specification Involved	Gages Rolled, inch	Destination
		250-Ksi	Grade Sheet		•
Republic 3321246	3321246	AM-Si/Al-DOX	RSC-AF-3A, C1 I	0.045 0.160	Aerojet-General
	3920851*	Si/Al-DOX-VAR	RSC-AF SA, C1 II	o.045 ° 0.160	Aerojet-General
Republic	3321262	D-C-DOX	<b></b>	••	Phase III
3321262	3920852*	VAR-C-DOX	RSC-AF-3A, C1 III	0, 045 0, 160	Aerojet-General
		200-Ksi	Grade Plate		•
Lebanon W 1443	3920795°	VAR-C-DOX	RSC-AF-2A, C1 III	0.375 0.750 3.0	Aerojet-General
Standard Steel E-5565	3920835*	Si/A1-DOX-VAR	RSC-AF-2A, C1 II	0,375 0,750 3,0	Aerojet-General
	E-5565	AM-\$i/Al-DOX	RSC-AF-2A, C1 I	0.375 0.750 3.0	Aerojet-General

Remelted by Republic Steel Corporation.

### Testing Conditions

Rolling studies were conducted at 1850 F, 1950 F, and 2050 F to determine optimum rolling temperatures and cross-rolling ratios for 9Ni-4Co (200) plate. 3/8-inch plate specimens were heat treated as follows before their mechanical and fracture-toughness properties were determined:

Normalize at 1650 F for 1 hr, air cool

Austenitize at 1550 F for 1 hr, oil quench

Double temper at 1000 F for 2 hr each, air cool.

The plates were rolled from forged slibs 4 inches thick.

Rolling studies were also conducted to determine optimum rolling temperatures and cross rolling ratios for 9Ni-4Co (250) sheet. Specimens were heat treated as follows before their mechanical and fracture-toughness properties were determined:

Normalize at 1650 F for 1 hr, air cool

Austenitize at 1475 F for 1 hr, oil quench

Refrigerate at -100 F for 2 hr

Double temper at 400 F for 2 hr each, air cool.

The specimens and testing conditions employed for evaluating the effects of processing conditions were identical with those used for the 18Ni maraging steels, described above.

## Hot-Rolling Studies - 3/8-Inch Plate

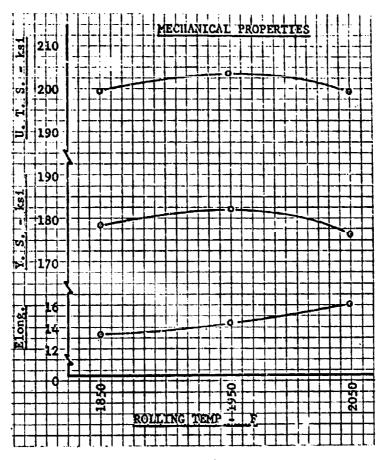
Figures 28 and 29 indicate that the tensile properties of 9Ni-4Co (200) steel are relatively insensitive to variations in rolling temperature. The vacuum-arc-remelted steel, however, exhibited far better fracture-toughness values than the air-melted in t (Heat No. E5565). The properties of the third heat evaluated (Heat No. 3920835) were intermediate between those shown in the two charts.

Based on these studies, 1950 F was chosen as the optimum temperature for rolling 9Ni-4Co plate and sheet bar. The data indicate that this practice provides a reasonable assurance of meeting the specification requirements for mechanical properties.

### Hot-Rolling Studies - 0, 160-Inch Sheet

The studies on effects of sheet-rolling temperature on properties of the 9Ni-4Co (250) steel were made on stock from three heats that were expected to develop yield strengths of 250 ksi. The effects of nine combinations of sheet bar and sheet-rolling temperatures are summarized in Table 32. The sheet-bar gage was 0.4 inch.

Table 32 shows that the fracture-toughness values of sheet from the air-melted heat (Heat No. 3321246) were poorer than those for vacuum-arc-remelted steel. The properties of the 9Ni-4Co (250) sheet produced from sheet bar rolled at 1850 F were



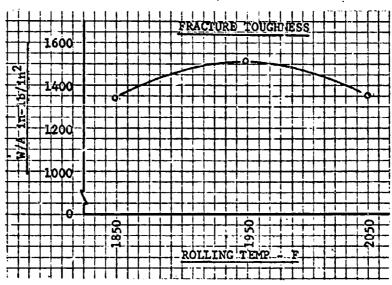


FIGURE 28. THE EFFECT OF ROLLING TEMPERATURE ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 9Ni-4Co(200)AM-Si/Al-DOX, HEAT E5565

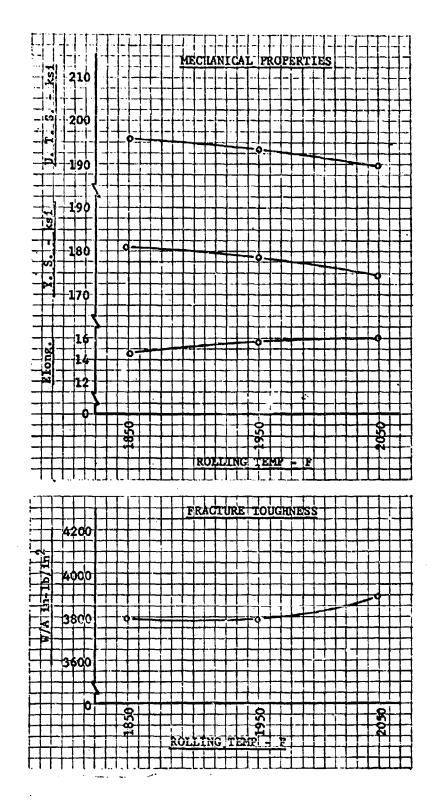


FIGURE 29. THE EFFECT OF ROLLING TEMPERATURE ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 9Ni-4Co(200)VAR-C-DOX, HEAT 3920795

TABLE 32. EFFECT OF ROLLING TEMPERATURE ON PROPERTIES OF 0, 160-INCH-THICK SHEET OF 9Ni-4Co (250) STEEL

Code Number	Kolling Temperature, F	Tensile Strength, ksi	0,20 Percent Yield Strength, ksi	Fracture Toughness, N/YS <sup>(a)</sup>
	Sheet E	Bar Rolled at	1850 F	
AM-D	1400	285.1	247. 5	0.307
Ht 3321246	1500	285.1	245, 2	0.293
	1600	284.1	243, 2	0. 276
Si/Al-DOX-VAR	1400	289.2	248.6	0. 265
Ht 3920851	1500	287.0	248.2	0.322
	1600	283.7	243.2	0.306
VAR-C-DOX	1400	287.8	243.5	0.300
Ht 3920352	1500	290.0	246. 1	0.340
	1600	278. 2	236.3	0.315
	Sheet I	Bar Rolled at	1950 F	
AM-D	1400	287.2	252, 8	0. 268
	1500	284.6	241.2	0.200
	1600	287.7	244.6	0.266
Si/Al-DOX-VAR	1400	287.0	243.6	0, 273
Ht 3920851	1500	288.0	250.6	0 287
	1600	295.2	254.7	0.262
VAR-D-DOX	1400	288.3	248.0	0.339
Ht 3920852	1500	282.1	239. 9	0, 326
	1600	282. 1	246. 6	0, 284
	Sheet I	Bar Rolled at	2050 F	
AM-D	1400	273.4	240.0	0, 13
	1500	288.6	251,8	0, 299
	1600	293.8	249.2	0.320
Si/Al-DOX-VAR	1400	282.9	250.7	0.288
Ht 3920851	1500	284.7	250.7 244.2	0.302
	1600	294.0	249.5	0, 272
WAR-C-DOX	1400	287.7	<b>243.5</b> §	0.301
Ht 3920852	1500	284.6	238. 2	0,321
	1600	281.8	235, 1	0.312

<sup>(</sup>a) Nominal strength of center-notched tensile specimen divided by the yield strength,

very consistent. This was true for all three steels investigated. Sheet produced from plate rolled at 1950 F or 2050 F exhibited a wider spread in mechanical properties.

Rolling sheet at 1500 F, from sheet bar rolled at 1850 F, produced the best mechanical properties in the 9Ni-4Co (250) VAR-C-DOX steel. That combination of rolling temperatures was also suitable for the other two steels.

Based on the data in Table 32, the optimum rolling temperatures for 9Ni-4Co (250) steel were:

Sheet-bar rolling 1850 F Sheet rolling 1500 F

It should be noted that 1950 F was the temperature recommended for rolling plate that was not to be rerolled to sheet. The conclusion that 1850 F is better for sheet bar was based largely on the desire to ensure consistent results on different lots of sheet.

### Cross-Rolling Studies

Slabs of 9Ni-4Co (200) steel 2-1/4-inches thick were rolled to 3/8-inch plate at 1950 F, with cross-rolling reductions of 0, 50, and 67 percent. Earlier work had indicated that 1950 F was the best rolling temperature for plate. The stock came from Heat Nos. 3920835 and 3920795, representing two variations of vacuum-arc-remelted steel. Figure 30 illustrates the results obtained for the latter heat. Yield and tensile strengths were relatively insensitive to cross rolling. This was also true for elongation values and for fracture-toughness values determined on longitudinal specimens. On the other hand, cross rolling improved the fracture-toughness values of samples taken perpendicular to the major rolling direction. Cross-rolling reductions of about 67 percent apparently minimize anisotropy of 9Ni-4Co plates.

To investigate the effect of cross rolling on mechanical properties of heat-treated sheet, 0.65-inch-thick sheet bar was rolled at 1550 F to 0.160-inch sheet. Sheets of the 9Ni-4Co (250) VAR-C-DOX and Si/Al-DOX VAR steel were reduced with cross-rolling reductions of 0, 50, and 67 percent.

So far as yield and tensile strengths are concerned, cross rolling 50 percent had no effect; none of the specimens were markedly anisotropic. The larger amount of transverse rolling improved the strength of longitudinal specimens, the changes ranging from 2 to 7 ksi.

The fracture-toughness values of sheets from the two heats did not show a consistent response to cross rolling. Increasing the amount of cross rolling lowered the N/YS values of longitudinal specimens of Heat No. 3920851 and raised those of transverse specimens of Heat No. 3920852. Cross rolling did not affect the fracture-toughness of specimens taken perpendicular to those just mentioned. Thus, it appears that cross-rolling reductions of 50 percent resulted in a minimum amount of mechanical anisotropy.

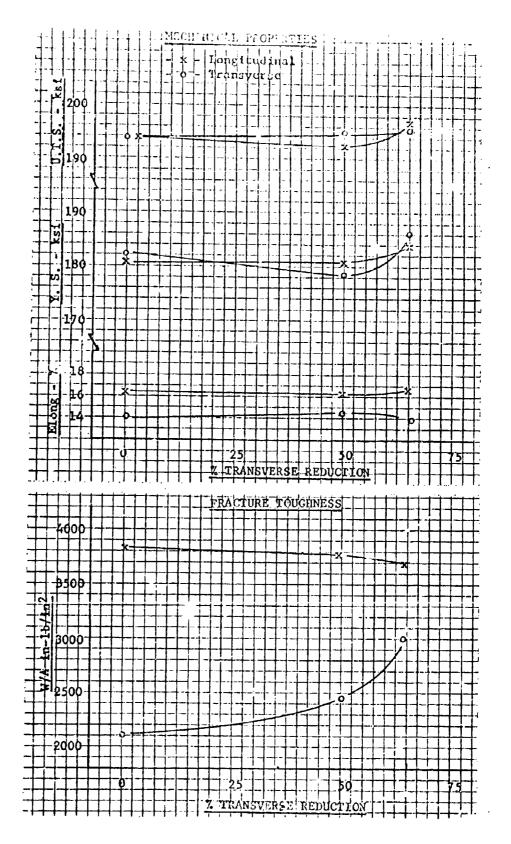


FIGURE 30. THE EFFECT OF CROSS ROLLING ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 9Ni-4Co(200)VAR-C-DOX, HEAT 3920795

## Heat Treatment Studies - 3/8, 3/4-Inch Plate

The 9Ni-4Co (200) steel is intended for use at the 170 to 180-ksi yield-strength level. That strength can be obtained by tempering at 950, 1000, or 1050 F. The current practice of using two 2-hour tempering treatments gives reproducible results. However, it would be more economical to use a single 4-hour treatment. Therefore, the effects of the two tempering practices were evaluated on samples of 3/8-inch plate.

All plate specimens were austenitized at 1550 F for 1 hour and then oil quenched. Specimens allocated for double-tempering treatments were treated at 950, or 1000, or 1050 F, each for 2 + 2 hours, followed by air cooling. Specimens allocated for single tempering were heated at 950, or 1000, or 1050 F, each for 4 hours, followed by air cooling.

The experimental plates represented Heat Nos. 3920835, E5565, and 392075. Differences in tensile and fracture toughness attributable to melting practice were detected. The ductility, impact resistance, and fracture toughness of the VAR-C-DOX material was decidedly superior to the properties of the other steels. In general, the fracture toughness of these 9Ni-4Co steels was lower than reported by earlier investigators.

Figure 31 shows the effect of different tempering treatments on the mechanical properties of the VAR-C-DOX steel (Heat No. 3920795). The Si/Al-DOX-VAR steel (Heat No. 3920835) responded similarly. The chart showed that double tempering at 1000 F developed the highest yield strength, about 10 ksi better than other treatments. It also gave the best fracture-toughness values. The W/A plateau for double tempering was about 3700 in. -lb/in. 2, compared to about 3400 in. -lb/in. 2 for the single-temper treatment.

The response of 3/4-inch plate samples from Heat No. 3920795 to various tempering schedules is shown by the data in Table 33. The tensile strength decreased gradually and regularly with increasing tempering temperature up to 1200 F. Tempering at 1100 F or lower temperatures, however, had no effect on the yield strength. Tempering at 400 or 600 F did not improve the Charpy values in comparison to those for as-quenched specimens, but higher temperatures were beneficial. The fracture-toughness values for fatigue-cracked Charpy specimens increased gradually with tempering temperature in the range up to 1100 F and then dropped. Variations in tempering time, or between single and double treatments, did not have significant effects on mechanical properties. Both types of notched bars indicated that specimens taken in the longitudinal direction were tougher than transverse specimens.

#### Heat-Treatment Studies - 0, 160-Inch Sheet

The 9Ni-4Co type of steel can be heat treated to either a martensitic or a bainitic microstructure. Many investigators had reported that the bainitic structure exhibits better fracture toughness than martensitic steels with equal strengths. Therefore, both types of heat treatment were investigated on sheet 0.160-inch thick.

All specimens for the study received the following preliminary heat treatments:

(1) Normalized at 1650 F for 1 hr, air cooled

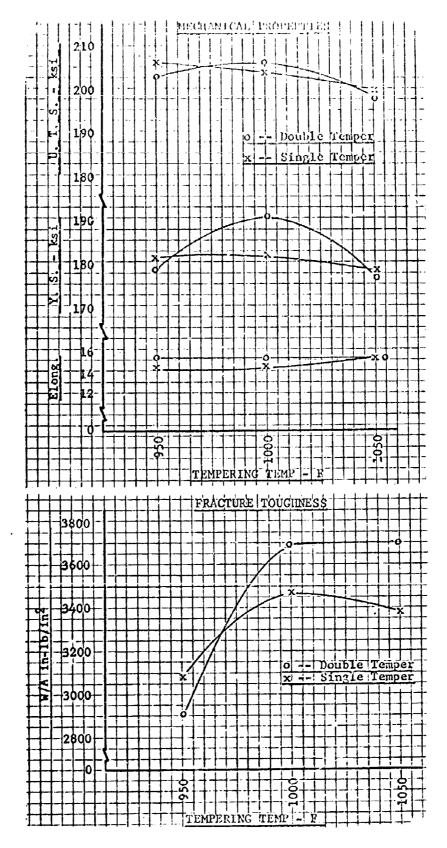


FIGURE 31. THE EFFECT OF TEMPERING TEMPERATURE AND TEMPER MODE ON THE MECHANICAL AND FRACTURE-TOUCHNESS PROPERTIES OF 3/8-INCH PLATE IN HEAT-TREATED 9Ni-4Co(200)VAR-C-DOX, HEAT 3920795

115

TABLE 33. RESPONSE OF SAMPLES FROM 3/4-INCH PLATES OF 9NI-4Co (200) VAR-C-DOX ALLOY STEEL (HEAT NO. 3/2073:) TO VARIOUS TEMPERING TREATMENTS

					:		,	i		Charpy V-Notch Value	rpy i Value	Fractute Toughness	ute iness
Temper Treatment, F	atment, F	Yield Su ksi	rength, si	Ultimate	Ultimate Tensile Strength, ksi	Area, percent	ton in sercent	Liongation, percent	ent ent	(koom 1 cmp), ft-1b	1 cmp),	inlb/in.	/in. 2
Temp, F	Duration	Т	1	T		Ţ	1	Ţ	ı	J.	1	1	   
None	اد	194.3	187.5	268,5	270.0	49.8	61.0	12.0	16. ¢	24.5	31,0	1210	3545
400	1 hr only	198.0	197.7	240, 1	240.6	. 53.5	63.3	12.5	14.0	28.7	37.5	1790	2235
400 + 400	2 hr each	193.6	193.0	237.2	236.5	57,1	82.2	13.0	14.5	30.0	35.0	1915	2120
400	4 hr	191.5	102.4	233,5	235,2	49.4	63.9	13,0	14.0	29.7	35, 7	1885	2190
009	1 hr only	189.3	189.7	216.4	217.2	57.3	<b>4.</b> 2	12.0	13.0	26.0	31.7	1770	2200
009 + 009	2 hr each	191.0	191.2	215,0	218.3	55.0	62.4	12.0	13.5	25.5	30.7	1838	2240
009	4 hr	188.0	185.7	215.8	215,7	55.0	<b>2</b> 0	12.0	13,5	25.3	31.0	1700	2190
800	1 hr calv	181.3	183.2	202.2	203.7	56.3	63.5	13.0	14.0	29.7	35,0	206.5	2480
800 + 800	2 hr cach	185,5	187.0	200.9	202.9	58.0	64.3	13.0	15,5	28.5	35,3	2175	2315
800	# # #	184.5	185.0	202.4	202.9	55.0	63.4	12.0	15.0	28.0	34.5	2150	01 03 03 03
1000	1 hr only	0.06.	189."	203.0	203.2	56.5	58.4	13.0	17.5	35.0	45.3	2560	3040
1000 + 1000	2 hr each	194.0	193.5	204.0	202.5	58.8	69.3	14.0	16.0	36.7	46.2	2160	3310
1000	4 14	190.5	190.2	202,5	201.2	54.3	68.8	14.0	16.0	36.5	46.2	2000	3360
1100.	1 hr only	183.7	180, 0	194,1	193.4	59.8	68.7	15.0	17.0	4.0	55.3	3350	3925
1100 + 1100	2 hr cach	181.0	184.0	187.5	193.0	57.6	69.2	15.0	17.5	41.0	56.5	3360	4225
1100	4 hr	175.7	176.1	186,2	186,1	59.3	69.1	15,5	18.0	49.0	63.5	3625	4020
1200	1 hr only	116.3	119.3	187.0	187.2	55.2	61.8	18.	18.0	45.0	51.3	3005	3340
1200 + 1200	2 hr cach	128.8	108.7	173.6	178.4	60.1	60,5	19.0	15.0	41.0	44.0	3610	4215
1200	4 hr	109.3	110.3	181.7	183.1	50.4	55.2	16.5	18.0	37.2	41.5	2525	04.55
All walness	(a) All malace and assessment for dualicate de	dualicate d	etermin ations	S.									

(a) All values are averages for duplicate determinations. (b) Hea. Treatment: Normalize, 1650 F I hour, sir cool;

Austenitize, 1550 F 1 hour, oil quench;

Temper, as indicated.

(c) Specimens 0,252-in, diameter Standard tensile bars

0.394 x 0.394 V-notch Charpy bars 0.394 x 0.394 V-notch Charpy bars prectacked to 0.020 to 0.040-inch crack depth.

- (2) Austenitized at 1475 F for 1 hr, oil quenched for martensitic specimens
- (3) Austenitized at 1475 F for 1 hr, quenched in salt at a temperature appropriate for bainitic specimens.

## Martensitic Specimens

The martensitic specimens were double tempered, 2 plus 2 hours, at 400, 450, or 700 F.

Figure 32 shows, as would be expected, that the strength of the 9Ni-4Co steel decreases as the tempering temperature is raised. The change amounts to about 10 ksi in yield or tensile strength for a 50 F change in tempering temperature. The yield strength of the other steel investigated (Heat No. 3920851) was about 10 ksi higher than that shown for the VAR-C-DOX steel after tempering at 400 or 450 F.

Tempering at higher temperatures improved the fracture toughness. The N/YS values shown in Figure 32 are much better than those obtained on the Si/Al-DOX-VAR steel (Heat No. 3920801), which ranged from 0.295 to 0.380.

## Bainitic Specimens

The bainitic specimens were transformed isothermally by holding 8 hours at 450, 475, or 500 F, or by holding 6 hours at 475 or 500 F. Increasing the transformation time from 6 to 8 hours had no effect on the yield strength of Heat No. 3920852 but lowered that of Heat No. 3920851 by about 10 ksi. No effect of transformation time on tensile strength was detected in either material. The effect of transformation temperature on fracture toughness shown in Figure 33 is typical of that found for both materials.

As in the case of martensitic specimens, Heat No. 3920852 had better N/YS values than Heat No. 3920851,

The tensile properties corresponding to the three transformation temperatures investigated showed a distinct advantage in favor of the 475 F temperature for both steels. The 230-ksi nominal yield strength attained at this transformation temperature was 10 to 15 ksi above the nominal yield strengths attained at transformation temperatures of 450 and 500 F. The tensile strength at all transformation temperatures was 260 ksi or higher.

The fracture-toughness-test results of the specimens transformed for 8 hours followed the inverse strength-to-toughness relationship. As with the martensitic heat treatment, a more pronounced effect of transformation temperature on toughness was observed in the VAR-C-DOX material. N/YS values varied from a minimum of 0.700, corresponding to the 475 F transformation temperature, to 0.780, corresponding to the 450 F transformation temperature. The N/YS values of the Si/Al-DOX-VAR steel varied from a minimum of 0.500, corresponding to 475 F bainite, to 0.540, corresponding to 500 F bainite. Fracture-toughness-test results of the specimens transformed for 6 hours at 475 and 500 F were not in accord with the results obtained for specimens transformed for 8 hours at the corresponding temperatures. The N/YS values in this

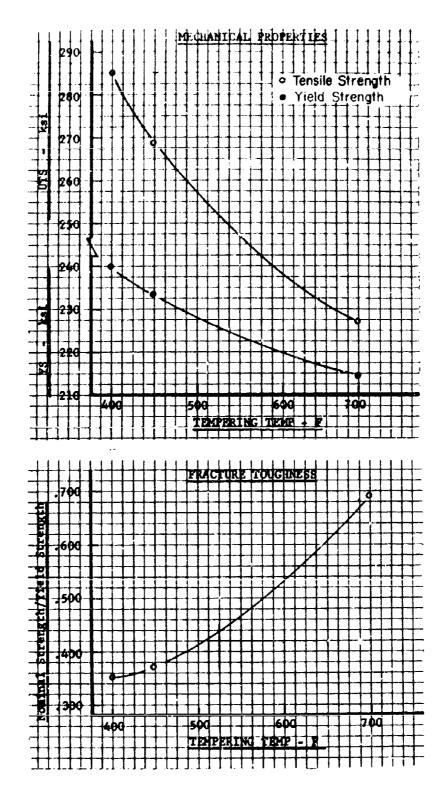
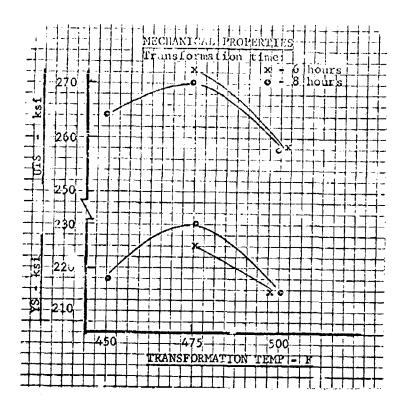


FIGURE 32. THE EFFECT OF TEMPERING TEMPERATURE ON THE MECHANICAL AND FRACTURE TOUGHNESS PROPERTIES OF 0.160-INCH-SHEET IN HEAT-TREATED 9Ni-4Co(250)VAR-C-DOX, HEAT 3920852



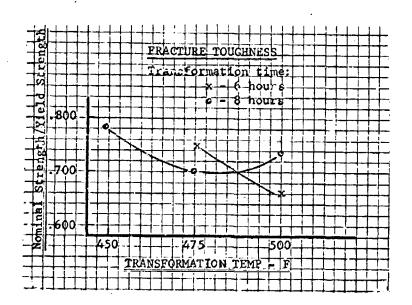


FIGURE 33. THE EFFECT OF TRANSFORMATION TEMPERATURE AND TIME ON THE MECHANICAL AND FRACTURE-TOUGHNESS PROPERTIES OF 0. 160-INCH SHEET IN HEAT-TREATED 9Ni-4Co(250)VAR-C-DOX, HEAT 3920852

case varied cirectly as the strength, as opposed to inversely, as shown above. The N/YS values of the specimens transformed for 6 hours at 475 F were 0, 100 unit above those determined in specimens with 10-ksi-lower yield strengths transformed for 6 hours at 500 F.

Bainite Versus Martensite. Figures 34 and 35 clearly substantiate the claims of improved fracture toughness with the bainitic heat treatment. These figures show plots of yield strength and tensile strength versus fracture toughness for both the martensitic and bainitic heat treatments. For the VAR-C-DOX material, it may be seen that a minimum advantage of 0.250 N/YS unit is attained at all strength levels with the bainitic heat treatment. A lesser improvement was seen in the Si/Al-DOX-VAR material. As shown above, this material was less responsive to variations in heat treatment and, in general, attained lower fracture-toughness levels.

## Evaluation of 18Ni Steel Plate and Sheet

## Prior History

The 18Ni (250) maraging steel was supplied to Aerojet-General Corporation as 0.045-inch and 0.160-inch sheet and as 3/8-inch, 3/4-inch, and 3.0-inch plate. The steels came from air-melted Heat No. 53088 and vacuum-arc-remelted Heat No. 3920864. In addition, sheets from vacuum-arc-remelted Heat No 08749 were also evaluated. The producers of the heats are indicated in Table 34. All stock was received in the solution-treated condition. The specimens were aged by Aerojet-General for 3 hours at 925 ±5 F in a resistance-heated vacuum furnace. That aging treatment was chosen on the basis of experiments previously conducted by Republic Steel Corporation. All data in this section of the report were obtained by Aerojet-General Corporation.

	OR EVALUATION OF 18N1 STEELS

Heat		Melting		
Number	Producer	Practice	Grade	Product Thickness, inch
3321290	Republic Steel Corporation	AM-D	250	3.0
08749	Vanadium Alloy Steel Company	VAR	300	0,045,0,160
3920864	Republic Steel Corporation	VAR	250	0,045,0,160,0.375,0.750,3.0
53088	United States Steel Corporation	MA	250	0.045, 0.160, 0.375, 0.750
604	Lukens	VAR	200	0.60 to 0.75
3920556	Republic Steel Corporation	VAR	200	0.60 to 0.75

## Tensile Properties

Tensile properties of sheet and plate as determined on longitudinal and transverse specimens are summarized in Table 35. Threaded round specimens were taken from the 3/4- and 3-inch plates; pin loaded specimens were used for the other materials. The variation in strengths measured in the longitudinal and transverse directions was small for the vacuum-arc-remelted steels, but noticeable in sheet from the air-melted heat. No consistent effect of melting practice on the level of tensile properties was observed in the 18Ni (250) maraging steels.

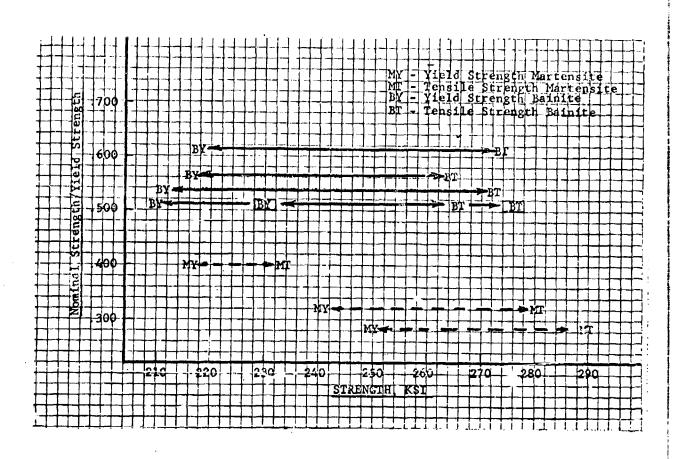


FIGURE 34. MARTENSITE VERSUS BAINITE: THE RELATIONSHIP OF STRENGTH TO FRACTURE TOUGHNESS IN BAINITIC AND MARTENSITIC MICROSTRUCTURES OF 0.160-INCH SHEET IN HEAT-TREATED 9Ni-4Co(250) Si/Al-DOX-VAR, HEAT 3920851

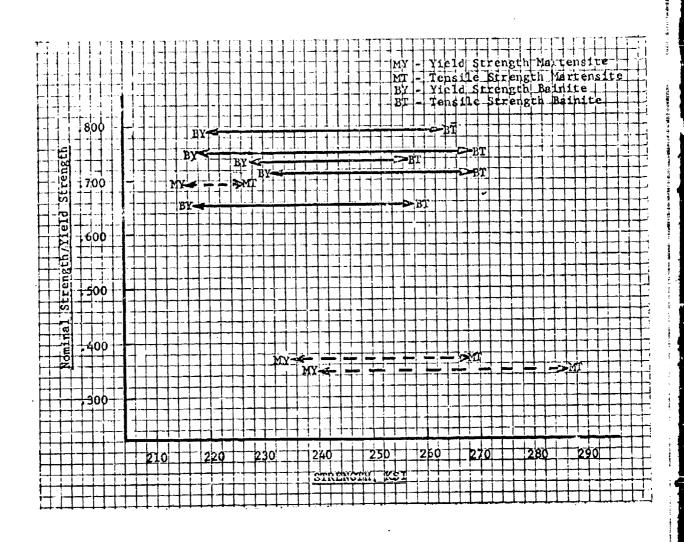


FIGURE 35. MARTENSITE VERSUS BAINITE: THE RELATIONSHIP OF STRENGTH TO FRACTURE TOUGHNESS IN BAINITIC AND MARTENSITIC MICROSTRUCTURES OF 0.160-INCH GAGE SHLET IN HEAT-TREATED 9Ni-4Co(250)VAR-C-DOX, HEAT 3920852

TABLE 35. TENSILE PROPERTIES OF HEAT -TREATED 18NI SHEET AND PLATE(4)

	:			Ultimat	Ultimate Strength,	0.2 Pe	0.2 Percent Yield			Red	Reduction of
Thic: nex,	Heat	Ç	Melting	1	ksi	Street	Strength, kg	Elongario	Elongation, percent	Are	Area, percent
IIICII	Number	Grade	Fractice	1	T	7	T	J	H	-3	H
0.045	08748	300	VAR	279.1	280.6	278.9	280.0	1.4	1.1	:	:
0.045	3920864	250	VAR	268.3	270.8	268.3	270.1	1.2	1,5		
0.045	53088	250	WV	234,4	246,8	234.4	245.9	1.3	<b>∞</b> −i		
0.160	08749	300	VAR	272.2	279.0	265.2	270.4	8.7	8.7		
0.160	3920864	250	VAR	260.8	262.5	248.5	250, 6	10,1	10.4		
0.160	53088	250	Ч	238.6	242.3	225,6	230.4	11.6	10.8		
0.375	A8749	300	VAR	. 1	ŀ	:	:	;	:		
0.375	3920664	250	VAR	263.2	264.1	254.0	254.3	20.0	17.5		
c. 375	53088	250	МА	239.5	239, 5	282.5	231.0	18.0	17.5		
0.750	08749	300	VAR	:	:	:	:	i	!		
0.750	3920864	250	VAR	248.4	251,8	225.4	237.4	12. 36)	10. 1 <b>(b)</b>	53.6	<b>4</b> .0
0,750	53088	250	Y.	244.0	237.4	226.4	227.0	9°6	12.20)	37.6	49,3
3.0	3321290	250	G-MA	254.3	255.2 250,5(c)	243.9	2 <b>41.</b> 8 242.7(c)	14.4	9.3 6.1 <sup>(c)</sup>	50, 4	38.2 28.5 <sup>(c)</sup>
3.0	3920864	250	VAR	243.8	242.3 241.3 <sup>(c)</sup>	233, 5	230, 6 232, 7(c)	11,8	9.3 5.2(c)	2.2	40.1 14.0(c)

<sup>(</sup>a) L = Jongmudana. and 1 = transverze spe (b) 2-inch gage length. (c) Short transverse (1-inch gage length).

The 0.045-inch sheet exhibited a higher yield strength and lower ductility than the 0.160-inch sheet from the same heat. Since this effect was noticed at both strength levels, it probably results from the differences in specimen thickness rather than by rolling history. The strength of the air-melted steel (250) was constant over the remaining range of thicknesses, 0.160 inch to 0.750 inch, investigated. The strength of the VAR (250) steel (Heat No. 3920864) was constant for the 0.160-inch and 0.375-inch flat-rolled stock, but dropped for the two heavier gages (0.75 inch and 3.0 inches). The strengths of the latter two products were comparable to those of the air-melted steel. The ductility was poorer for transverse and short-transverse specimens from the 3-inch plate, compared with longitudinal specimens or with thinner plate.

Table 36 gives the tensile properties of two 18Ni (200) heats of maraging steel Heat Nos. 604 and 3920556.\* The 0.2 percent offset yield strengths and ductility values are similar to those in Table 35 for the 18Ni (250) material. Both heats had slightly higher strengths in the transverse direction, but the differences may not be significant from an engineering standpoint.

TABLE 36. TENSILE PROPERTIES OF HEAT TREATED 18N1 (200) 0.65-INCH STEEL PLATE(a)

Heat Number	Specimen Orientation	Ultimate Tensile Strength, ksi	0.20 Percent <sup>(b)</sup> Yield Strength, ksi	Percent Elongation(c)	Reduction in Area, percent
Lukens 604, VAR	Longitudinal	235.2	229.0	14	57.6
	Transverse	239.0	<b>232.</b> 0	12	53,2
3920550, VAR	Longitudinal	228.0	218.0	11.3	61.3
	Transverse	234.0	221.0	11.6	59.1

<sup>(</sup>a) These plates were 0.6 to 0.750-inch thick.

#### Charpy Properties

The results of tests on standard Charpy V-notch specimens at various temperatures are given in Table 37. The samples were taken from plates representing two heats of 18Ni (250) steel. As is customary, the values were higher for longitudinal than for transverse specimens. Although the values decreased with testing temperature, the longitudinal specimens of the air-melted heat (Heat No. 53088) were the only ones that exhibited a ductile-brittle transition. The data for longitudinal specimens indicate a transition temperature between 80 and 200 F. These data indicate that melting practice does not have a significant effect on Charpy properties. This agrees with data on specimens taken from forgings and discussed earlier in the report.

<sup>(</sup>b) Supplied in the solution-treated condition. Aged at 900 F for 8 hours.

<sup>(</sup>c) 1-inch gage length.

These hears were produced on Contract AF 04(695)-350.

TABLE 37. CHARPY IMPACT PROPERTIES OF HEAT-TREATED 18N1 PLATE

Thickness,	Heat		Melting	Specimen	C	harpy-I	npact S	trength a	t Indicate	d Tempe	rature, fs	-lb
inch	Number	Grade	Practice	Orientation	-80 F	0 F	RT	200 F	300 F	375 F	400 F	500 P
0.750	3920864	250	VAR	L	12,4	13,8	17.0	17.6	22	~=	25, 9	25.7
				T	10.0	12.1	13.1	17,2	16.6		17.5	18.2
0.750	53088	250	AM	L	12.0		11.9	17.6	19, 8		22,9	20.3
		٠.		T	10.6	••	10.0	12.3	12.5		13,7	16.8
3.0	3920864	250	VAR	L	15.4	17.8	17.8	22.4				
				Т	12.6	12.0	13,7	14, 1	••	17.6		
3,0	<b>33212</b> 90	250	AM-D	L	13.8	16.4	16.7	18.4	•		**	
				T	12.3	12.8	15.0	13.6				

## Precracked-Charpy-Impact Properties

Precracked Charpy specimens of the type described previously were also used to evaluate the maraging steels. The results are compared in Table 38 with data obtained on Heat Nos. 604 and 3920556 produced for another contract (AF 04(695)-350). Although the data for 18Ni (250) are scanty, the results are considered typical except for the abnormally high values for the longitudinal specimens from Heat No. 53088 tested at room temperature. Despite the fact that the 18Ni (200) steels had yield strengths comparable to 18Ni (250), they gave better fracture-toughness values. The W/A values of both 18Ni (200) steels increased with testing temperature.

TABLE 38. PRECRACKED CHARPY IMPACT PROPERTIES OF 18Ni STEELS

Heat		Melting	Specimen	0,2 Percent Yield	W/A	at Indic	ated Te	mperature	, in1b/	/in. 2
Number	Grade	Practice	Orientation	Strength, ksi	-100 F	-40 F	RT	+200 F	+300 F	+400 F
3920864	250 <sup>(a)</sup>	VAR	Ţ.	225			685	••		
			T	237			584			
58088	250(a)	AM	L	226		-	1044	797	1058	
			T	227			484	702	693	
604	200 <sup>(b)</sup>	VAR	L	229		813	988	1095		
			T	232	Ţ. <b>-</b>	635	800	928		
3920556	20n(b)	<b>VA</b> R	L	218	1028		1509	1502		2279
			T	221	893		1340	1417		2106

<sup>(</sup>a) From 0, 750-inch plate aged 3 hours at 925 ±5 F.

#### Environmental Effects

Fatigue-precracked tensile specimens of the type shown in Figure 36 were immersed in a 3 percent solution of NaCl in water at 70 F and subjected to sustained loads. The loads applied corresponded to 90, 80, and 70 percent of the notched tensile strengths of the steels determined in air for the same type of specimens. The failure times of the specimens are shown in Table 39. It was not established whether the delayed cracking resulted from stress corrosion or hydrogen embrittlement.

<sup>(</sup>b) Aged 8 hours at 900 F; Heat No. 604 made by Lukens; Heat No. 3920556 was made by Republic.

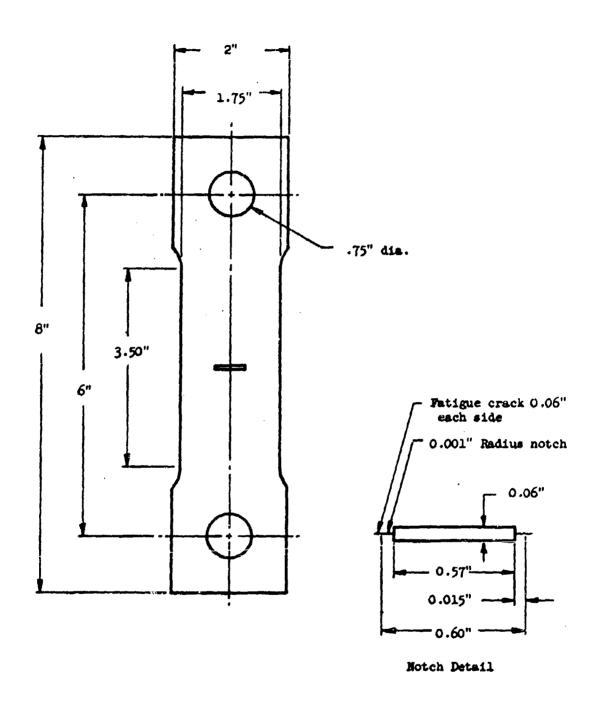


FIGURE 36. ENVIRONMENTAL TEST SPECIMEN FOR EVALUATING 0, 045 AND 0, 160-INCH-THICK SHEET MATERIAL

TABLE 39. FAILURE TIMES FOR FATIGUE-PRECRACKED TENSILE SPECIMEN OF 18N1 STEELS IMMERSED IN 3 PERCENT Nacl at 80 F

Heat Number (Sheet Thickness)	Grade	Melting Practice	0, 2 Percent Offset Yield Strength, ksi	Notch Tensile Strength, ksi	Percent of Notch Tensile Strength	Initial Applied Stress, ksi	Test Time, hr:min
08749	300	VAR	278.9	176,5	100		**
(0.045 in.)				189.0	100		
(					90	163.2	14:30
					90	162.4	35:21
					80	154.6	28:50
					80	144.3	33:50
					70	129.0	42:05
					70	124,8	<b>43:4</b> 5
3920864	250	VAR	268.3	184.2	<b>10</b> 0		
(0,045 in.)	4		-	200.4	100		
(0,015 1.1.)				· -	90	172.0	252.0 <sup>(b)</sup>
					90	167.2	26:35
					80	150.0	24:25
					80	155.8	<b>42:</b> 08
					70	131.0	48:00 <sup>(b)</sup>
					70	138.0	36:00
50.00	050		234, 4	182.8	100		
53088	250	AM	234,4	177.4	100		
(0.045 in.)				2116-	90	156.8	:01
					90	150.3	0
					80	142.9	138:15
						136.7	23:40
					80		44:36
					70 70	123.6 118.1	46:25
						•	
08749	300	V.AR	262.2	187.5	100 <b>9</b> 0	164.2	9:45
(0.160 in.)						169.9	23:20
•					90		
				÷	80	152.9	5:55 07:03
					80	151.7	27:23
					70	130.2	24:00
					70	131.0	31:57
3920864	250	VAR	248.5	178.6	100	~ =	**
(0.160 in.)				185.3	100	••	
					90	169.0	32:55
				•	90	167.5	30:10
					80	149.2	32:25
					80	142.6	44:10
					70	128.1	48 (b)
					70	131.1	48:(b)
<b>5308</b> 8	259	AM	225.6	189.3	100	* <b>-</b>	-,-
(0.160 in.)	-04	- 1272	=#V# V		90	170.9	:09
					90	171.9	:07
				•	80	157.0	18:15
					80	158.0	44:57
					70	143.9	33:29
					70	136.2	56;58

<sup>(</sup>a) Centerline of all specimens longitudinal to the sheet rolling direction. Specimen configuration shown in Figure 36.

<sup>(</sup>b) No failure in testing time indicated.

The data show that sheet specimens of the air-melted steel (Heat No. 53088) failed almost immediately when stressed at 90 percent of its notch tensile strength. Conversely, the specimens of vacuum-arc-remelted steel withstood equivalent stressing for appreciable time intervals. Thus, the investigation indicates that exposure to 3 percent NaCl solutions has less effect on steels made by vacuum-arc-remelting practices. The tests at 70 percent of the notch tensile strength disclosed no significant difference in failure times attributable to melting practice. Failure times increased as stress decreased.

The data suggest that maraging steels with lower strengths are more resistant to delayed brittle fracture, in dilute NaCl solutions, than those with higher strengths. This behavior pattern is typical for delayed hydrogen-stress cracking.

## Fracture Toughness

Three types of notched tensile specimens were used to evaluate the fracture toughness of the maraging steels. The test performance of the materials with different histories was judged by the following parameters:

K<sub>c</sub> = critical-stress-intensity factor associated with initiation of unstable plane-stress fracturing

K<sub>Ic</sub> = critical-stress-intensity factor associated with initiation of unstable plane-strain fracturing.

A center-notched specimen was used for determining the parameter  $K_{\rm C}$  for 0.045-inch and 0.160-inch-thick sheet. Cracks were produced at the ends of the center notch by low-cycle flexure at a maximum bending stress of half the yield strength. Pin loading was employed during tensile testing to minimize eccentric loading. An extensometer-type clip gage inserted into the center notch was used to measure the opening during loading. Calibration curves for the maraging steel (and for the 9Ni-4Co steels discussed later) were used in conjunction with test measurements to establish the load and crack extension at the time of instability. Those data were used with the following equation to calculate the plane-stress fracture-toughness parameter,  $K_{\rm C}$ :

$$K_c = F \left[ W \tan \frac{a\eta}{W} \right]^{1/2}$$

where

F = gross stress

W = specimen width

a = half-crack length.

The results of the center-notched tensile tests on the 18Ni steels are summarized in Table 40. Over half of the tests on the 0.160-inch-thick specimens and three of the four tests on the 0.045-inch sheet of air-melted steel (Heat No. 53088) were invalid by ASTM standards because the net stress developed during testing exceeded 80 percent of the yield strength. At least, qualitatively, however, the K<sub>c</sub> values indicated that the 18Ni (250) air-melted and vacuum-arc-remelted steels had comparable fracture-toughness properties. However, the yield strength of the latter steel was higher by about 25 ksi. The 18Ni (300) vacuum-arc-remelted steel had K<sub>c</sub> values about the same as

the other materials. In general, therefore, it appears that vacuum-arc remelting results in improved fracture toughness for maraging steels.

TABLE 40. FRACTURE-TOUGHNESS VALUES, K<sub>C</sub>, FOR LONGITUDINAL AND TRANSVERSE SPECIMENS OF 18N1 STEEL<sup>(a)</sup>

Sheet Thickness,			Melting	Yield S	Percent Offset Id Strength, ksi		K <sub>C</sub> Value, ksi √in,	
inch	Heat Nuniber	Grade	Practice	L	T	L	T	
0.045	08749	300	VAR	278.9	280.0	159	141	
0.045	3920864	250	VAR	268.3	270.1	151	147	
0.045	53088	250	AM	234.4	240.9	178	153	
0.160	08749	300	VAR	265.2	270.4	149	168	
0.160	3920864	250	VAR	248.5	250.6	186	178	
0.160	53088	250	AM	225.6	230.4	186	179	

(a) Determined on specimens of the type shown in Figure 22.

The plane-strain fracture toughness of the steels was evaluated by determining the parameter  $K_{\rm IC}$  on precracked tensile specimens. Figures 37, 38, and 39 show the types of specimens employed for studies on stock of different thicknesses up to 0.75 inch. These specimens were precracked by using an electrical-discharge machine to start the notch and then flexing the samples repeatedly with a stress of less than half the yield strength. The precracks produced by fatigue  $m_s t$  the following ASTM requirements for fracture testing:

- (1) Crack depth less than 50 percent of the specimen thickness
- (2) Crack area less than 10 percent of the specimen area.

During tensile testing, a load of 500 lb/sec was maintained. The load at failure and the original crack dimensions were used to calculate  $K_{Ic}$  from the following equation, which was developed by Irwin:

$$K_{Ic} = 1.21 \pi \frac{a}{Q} F^2 ,$$

where

a = depth of the fatigue crack

Q = flaw shape parameter

F = gross fracture stress.

In order to obtain quantitative values of the parameter for plane-strain fracture toughness, the gross fracture strength must be less than the 0.2 percent offset yield strength of the material.

Table 41 summarizes the results of the plane-strain fracture-toughness tests on the flat, precracked tensile specimens. The data on 0.160-inch sheet showed that the fracture toughness of the 18Ni (300) was poorer than that of the lower-strength 18Ni (250). Despite its higher yield strength (higher by 20 ksi), the K<sub>IC</sub> values for the VAR sheet were better than those for the air-melted steel.

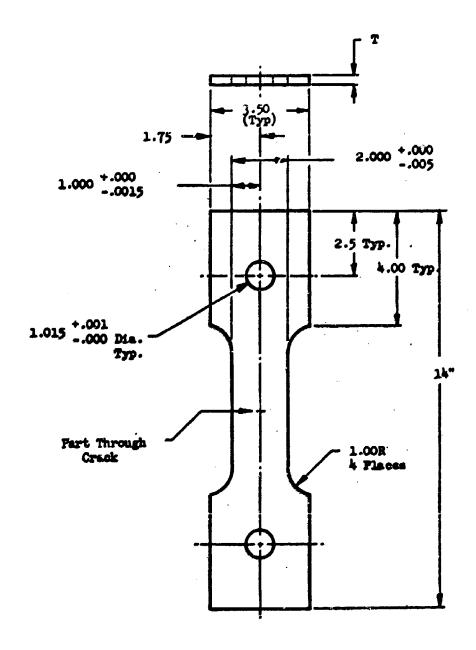
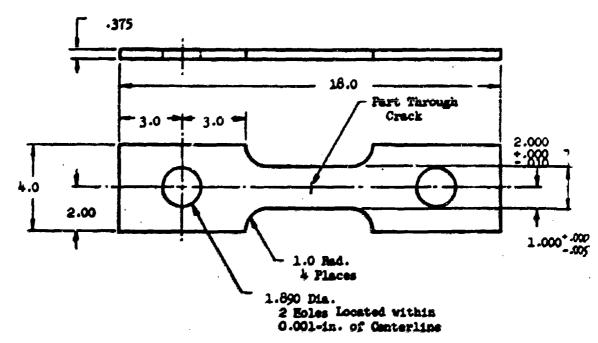


FIGURE 37. PART-THROUGH-CRACK (PTC) TENSILE SPECIMEN FOR EVALUATING 0. 160-INCH-THICK SHEET MATERIAL



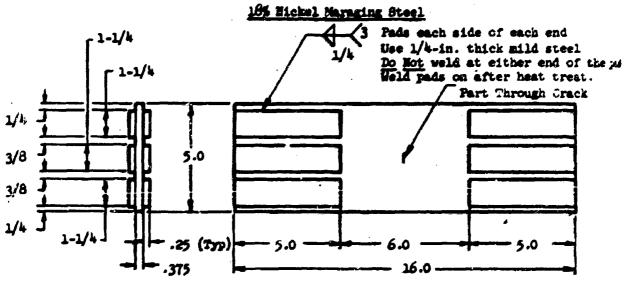
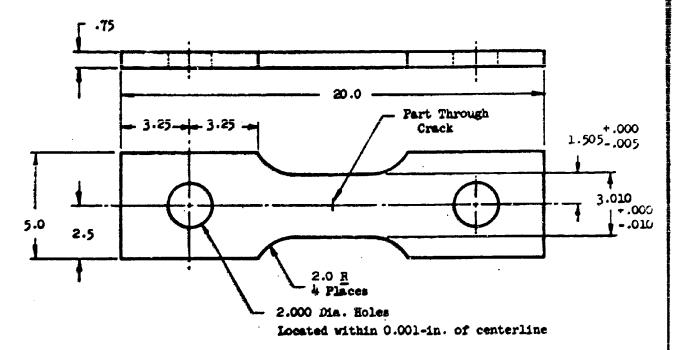


FIGURE 38. PART-THROUGH-CRACK (PTC) TENSILE-SPECIMEN CONFIGURATION FOR EVALUATING THE 3/8-INCH-THICK PLATE MATERIALS

95 Nickel - 45 Cobalt Steel



5% Nickel - 4% Cobalt Steel

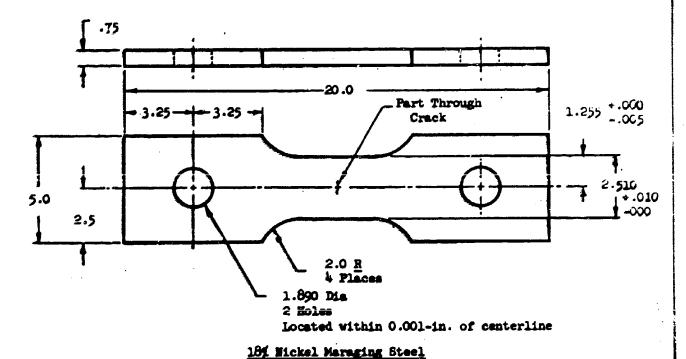


FIGURE 39. PART-THROUGH-CRACK (PTC) TENSILE-SPECIMEN CONFIGURATION FOR EVALUATING THE 3/4-INCH-THICK PLATE MATERIALS

TABLE 41. FRACTURE-TOUGHNESS VALUES,  $K_{\rm lc^*}$  FOR LOGITUDINAL AND TRANSVERSE SPECIMENS OF 18NI STEEL (4)

Specimen Thickness,			Melting	Yield St	ent Offset trength,		Value,
inch	Heat Number	Grade	Practice	L	T	L	T
0.160	08749	300	VAR		270.4		103, 87
0.160	3920864	250	VAR		250.5		114, 111
0.160	53088	250	MA		230.4		107, 103
0.375	3920864	250	VAR	254.0	254.3	104	99
0.375	63088	250	MA	232.6	231.0	94	97
0.750	3920864	250	VAR	225.4	237,4	104	100
0.750	53088	<b>2</b> 50	AM	226.4	227.0	102	113
0.6-0.75	604	200(b)	VAR		232.0		123, 114
0.6-6.75	3920556	200 <sup>(b)</sup>	AM		221.0		155

<sup>(</sup>a) Specimen configurations are shown in Figures 37, 38, and 39. The Grade 250 and 300 steels were aged 3 hours at 925 ±5 F.

In the thicker flat-rolled products, no consistent variation in fracture toughness was associated with specimen orientation. Their  $K_{\rm IC}$  values were similar to those determined on sheet specimens. The VAR (250) specimens generally had better  $K_{\rm IC}$  values than the samples of air-melted steel (Heat No. 53088). They also had higher yield strengths. Thus, the data confirm the general opinion that vacuum-arc remelting improves fracture toughness.

Table 41 also shows that two heats of 18Ni (200) steel had better  $K_{IC}$  values than the higher-strength grades, even though the actual yield strengths are not widely different. The lower-strength air-melted steel (Heat No. 3920556) had the best  $K_{IC}$  value of all specimens tested. These results suggest that superior fracture toughness is obtained, at an equivalent strength level, by aging a steel with lower titanium, molybdonum, and cobalt contents to its maximum strength than by underaging or overaging a higher-alloy material.

To obtain the plane-strain fracture-toughness value,  $K_{\rm IC}$ , for the 3-inch-thick maraging steels, specimens with the configuration indicated in Figure 40 were used. These round, notched specimens were fatigue precracked with a minimum-to-maximum tensile-stress ratio of 0.06 and a maximum stress of approximately 40 ksi. The ratio of the root diameter of the crack to the shank diameter of the specimen was controlled between 0.6 and 0.8. The loading rate for the notched tensile specimens was 100,000 lb/min. After failure, the diameter at the bottom of the notch was determined with an optical comparator. The failure load and the specimen and flaw dimensions were then used to calculate the  $K_{\rm IC}$  values from the following equation:

$$K_{Ic} = \frac{0.233 \text{ on } \sqrt{\pi D_0}}{\left[ - \left[ \frac{K_{Ic}^2}{2 \sigma_{VS}^2 \pi D_0} \right]^2} \right]}$$

<sup>(</sup>b) Aged 2 hours at 900 F.

where

 $\sigma_n$  = the net section stress

D<sub>0</sub> = the major diameter of the specimen

 $\sigma_{VS}$  = the 0.2 percent offset yield strength of the material.

In order to conform to ASTM requirements, the ratio of  $\sigma_n$  to  $\sigma_{ys}$  should not exceed 1.1.

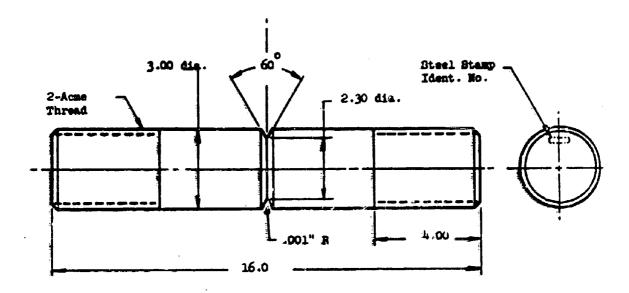


FIGURE 40. NOTCH TENSILE SPECIMEN FOR EVALUATING 3.0-INCH-THICK PLATE MATERIALS

Tests on round, notched tensile specimens from 3-inch plates of two 18Ni (250) maraging steel gave  $K_{IC}$  values considered valid according to ASTM requirements. The values, expressed in units of ksi  $\sqrt{\text{in.}}$ , were:

	Longitudinal	Transverse
Heat No. 3920864 (VAR)	<b>6</b> 9	61
Heat No. 3321290 (AM-D)	50	47

Although the longitudinal specimens gave higher values, the difference is not considered significant for the air-melted, degassed steel. The vacuum-arc-remelted steel, which had a yield strength higher by 10 ksi, exhibited better plane-strain fracture toughness.

It should be noted that the  $\rm K_{IC}$  values for the 3-inch plate from Heat No. 3920864 are much lower than those given in Table 42 for thinner plate. The differences, which are considered significant, are believed to indicate that differences in hot-rolling reductions can affect the toughness of 18Ni maraging steel. If this be true, the 3-inch plate received less than the critical reduction needed to develop the maximum planestrain fracture toughness in this type of steel. On the other hand, the Charpy test did not detect any differences between the 3/4- and the 3-inch plate.

TABLE 42. PRIOR HISTORY OF PLATE AND SHEET PRODUCTS USED FOR EVALUATION OF 9Ni-4Co STEELS

Heat Numbe <i>r</i>	Grade	Melting Practice	Product Thickness, inch	
3321246	250	AM-Si/Al-DOX	0,045,0,160	
3920851 250		Si/Al-DOX-VAR	0.045,0.160	
3920852	250	VAR-C-DOX	0. 045, 0. 160	
3920795	200	VAR-C-DOX	0.375,0.750,3.0	
3920835	200	Si/Al-DOX-VAR	0, 375, 0, 750, 3, 0	
E5565	200	AM-Si/Al-DOX 0, 375, 0.		

The tests on specimens from the 3-inch plate were conducted by the Aerospace Division of Boeing Company, Seattle.

# Metallographic Examinations

Metallographic examinations were conducted on samples taken from 3/8-, 3/4-, and 3-inch plates from Heat Nos. 3920864 (VAR) and 53088 (AM). The inclusion ratings of all products met the requirements of Specification RSC-AF-1 established for this program. No differences in inclusion ratings attributable to melting practice were detected except for samples from the 3-inch plates. For that product, the vacuum-arc remelted steel generally had lower inclusion ratings than the air-melted steel except for titanium nitrides. The inclusion ratings did not vary significantly with plate thickness.

The grain sizes of the 3/8-inch plate were uniformly ASTM 8 or finer for Heat No. 3920864 and predominantly 6 to 8 with some 4 for Heat No. 53088. The grain sizes of the 3/4-inch plate were comparable to the thinner plate but a little finer in the case of heat No. 53088. For both heats, the grain size of the 3-inch plate was predominantly ASTM 5 or finer.

The inclusion ratings and microstructures were independent of specimen orientation. Nor did they vary among samples from surface, mid-radius and center locations from the 3-inch plate.

To evaluate bonding, aged samples were etched with Marbles reagent. The metallographic examinations indicated the microstructures were typical of 18Ni maraging steel. For all plate thicknesses, banding existed to only a slight degree. Banding was a little more pronounced in the air-melted 3/8-inch plate. Some evidence of incomplete recrystallization was apparent in the 3/4-inch plate from the air-melted Heat No. 53088. In summary, the metallographic examinations detected no effects of differences in melting practice or plate thickness on microstructure.

#### Evaluation of 9Ni-4Co Steel Plate and Sheet

# Prior History

The stock for evaluation by Aerojet-General Corporation was received in three sheet gages of 9Ni-4Co (250) and three plate thicknesses of 9Ni-4Co (200) steel. The material came from six heats made by three different melting practices as indicated in Table 42. The 9N -4Co (250) steel contained 0.45 percent carbon; the 9Ni-4Co (200) steel contained 0.25 percent carbon. The steels were heat treated by the California-Doran Heat Treating Company, Los Angeles, California. Copper plating was employed to prevent carburizing, decarburizing, or oxidation of the specimens during the normalizing and austenitizing cycles.

The sheet specimens of 9Ni-4Co (250) steel were processed as a single group and then tempered as individual groups, depending on the heat number, as indicated below.

- (1) Electrolytically copper plate all parts a minimum plate thickness of 0.0005 inch prior to heat treatment.
- (2) Normalize at 1650 F for 1 hour at heat. Air cool to room temperature.
- (3) Austenitize at 1450 F for 1 hour at heat. Oil quench to room temperature (oil temperature: 70 to 100 F).
- (4) Refrigerate at minus 100 F for 2 hours and return to room temperature.
- (5) Temper for 2 hours at the following temperature for the indicated heat numbers:

Heat Number	Temper Temperature, F
3321246	425
3920851	<u>4</u> 25
3920852	400

- (6) Strip copper plate by alkaline cyanide method.
- (7) Perform the second temper for 2 hours at the temperatures indicated for the heats listed in Step 5 shown above.

The plate specimens 9Ni-4Co (200) steel were hardened and tempered as a single group according to the following procedure:

- (1) Electrolytically copper plate all parts a minimum of 0,0005 inch prior to heat treatment.
- (2) Normalize at 1650 F for 1 hour at heat.
- (3) Austenitize at 1500 F for 1 hour at heat. Oil quench to room temperature (oil temperature: 70 to 100 F).
- (4) Temper at 1000 F for 2 hours and air cool to room temperature,
- (5) Strip copper plate by alkaline cyanide method.
- (6) Perform second temper at 1000 F for 2 hours and air cool to room temperature.

# Tensile Properties

Table 43 lists the tensile properties of the 9Ni-4Co (250) sheet steel. The small differences in strength and ductility between products from different heats are not considered significant. Transverse specimens of 0.045-inch sheet of the VAR-C-DOX heat (Heat No. 3920852) had yield strengths about 9 ks. higher than those of longitudinal specimens. This anisotropy is attributed primarily to rolling history rather than to melting practice. The properties of all other materials were independent of specimen orientation. The strengths and elongation values of the 0.160-inch sheet were higher than those for thinner stock. These disparities are attributed partly to specimen geometry and partly to variations in hot-rolling practice.

TABLE 43. TENSILE PROPERTIES OF HEAT TREATED 9NI-4Co (250) SHEET

								E	ongation	n, perce	nt
Thickness,	Heat			Ultimate Tensile Strength, ksi		0.2 Percent Offset Strength, ksi		Ga	nch ige igth		nch ge gth
inch	Number	Grade	Melting Practice	L	T	L	T	L	T	L	Ť
0,045	3321246	250	AM ·SI/AI-DOX	282.2	276.2	236.9	232.0	7.1	6.8	5, 1	4.7
	3920851	250	Si/Al-DOX-VAR	278.8	282.0	233.6	235.1	7.4	6.9	4.9	4.6
	3920852	250	VAR-C-DOX	282,1	286, 1	227.6	236.9	7.1	7.3	4.6	5, 2
0.160	3321246	250	AM-Si/Al-DOX	288.9	288, 1	238.4	238.4			7.3	7.1
	3920851	250	SI/AI-DOX-VAR	289.9	289,6	240.6	240.8			7.3	7.8
	3920852	250	VAR-C-DOX	285, 4	286.6	234.3	237.8			7.4	8, 1

Table 44 summarizes the tensile properties of the 9Ni-4Co (200) plate steels. The data show that the properties were independent of plate thickness. Although strength values were almost identical for specimens with different orientations, ductility values were better for longitudinal than for transverse specimens. The ductility of the 3-inch plate was poorer in the short-transverse than in the transverse direction. Heat 390795 had lower strengths, and better ductility, than the other two heats but the differences were probably not caused by melting practice. Comparison of data for Heat Nos. E5565 and 3920835, which had comparable strengths, indicates that vacuum-arc remelting conferred a slight improvement in ductility on the 9Ni-4Co (200) steel.

#### Charpy Properties

Table 45 lists the standard Charpy V-notch values of samples from the 9Ni-4Co (200) steel plates. The VAR-C-DOX heat gave the best values in the Charpy test; they decreased gradually with testing temperature over the range investigated. With few exceptions, the Charpy values were low for samples of Heat No. 5565 (AM-Si/Al-DOX) tested at or below room temperature. Steel 3920835 (Si/Al-DOX-VAR) appeared to have a brittle-ductile transition temperature between 0 and 80 F. With the exception of Heat No. 5565, the Charpy values of the 9Ni-4Co (200) steels, with yield strengths approximating 183 ksi, were better than those for the 18Ni maraging steels, with yield strengths around 240 ksi, shown in Table 37.

Specimen orientation had a small but consistent effect; the Charpy values of transverse specimens were poorer. Vacuum-arc remelting improved notched-bar toughness.

TABL: 44. TENSILE PROPERTIES OF HEAT TREATED ?NI-4Co (200) PLATE

Thickness, Hest inch Number 0.375 F5565				•			į	Elongation, percent	, percent			
			Ultima	Ultimate Tensile Strength, ksi	O. 20 Per	0.20 Percent Offset Yield Strength, lai	1-In	1-inch Gage Length	2-Inch G	2-Inch Gage Length	Redu	Reduction in
	r Grade	Melting Practice	د	Ţ	Г	H	ı	F	-1	-	1	L
		AM-Si/Al-DOX	201.9	8 006	184 4	8						
300000	×	444. 704.17.3	,	•		707	7.07	7.77	15.2	14.8	:	;
80366	202	SI/AI-DOX-VAR	201.8	201.2	184.8	182.9	27.9	25.5	16.4	16.6	;	;
3920795		VAR-C-DOX	195.7	194.1	184.1	181.7	30.2	27.5	17.2	16.1	;	;
750				;	,							
	2003	AM-SI/AI-DOX	203.8	203, 5	183.3	182.9	•	:	15.8	14.8	48.7	42.7
3920835		Si/A1-DOX-VAR	202.9	203,3	184.2	185.5	;	:	18.	16.9		
390,0795		WAD-C-TANK	5						107	0.01	- 00	7.50
		VAR- 2-100	5°/87	195.9	18%	181,8	:	:	18.9	16,4	<b>6</b> 5, 1	£.3
3.0 E5565	006	AM-C(/A)-MA	3	9								
		VOT-14/16- WW	5	202.0	180.6	182.3	:	:	16.0	14.4	47,3	41.9
			:	202.34	•	182, 2(4)	:	13.2(a)	;	:	;	(e) 5.
3350832	200	SI/AI-DOX-VAR	203.6	203.6	183.9	183.7	ł	:	17.0	16.9	5.3	5.3.7
			:	203,2(4)	;	184, 4(4)	:	15.0(8)	:	:		(4)
3920795	200	VAR-C-DOX	194.0	194.0	180.0	180.2	;	;	19.0	6 61	3	
			ļ	(T) (T)				(4)	2	•	į	00.00
				130.67	:	179.04"	:	15.4	:	;	;	53.4(2)

138

TABLE 45. CHARPY IMPACT PROPERTIES OF HEAT-TREATED 9NI-4Co (200) PLATE

Thickness	Heat		Specimen	0.20 Percent Offset Yield	Charpy	Impact		at Indicat	ed Temp	erature,
inch	Number	Melting Practice	Orientation	Strength, ksi	-110 F	-80 <b>F</b>	-40 F	0 F	RT	+200 F
0.750	E5565	AM-SI/AI-DOX	L	*=	9.5	8.7	10.5	11.8	13.3	20.3
			T		7,8	9, 1	8.5	9.6	13.2	16.2
	3920835	SI/A1-DOX-VAR	L		9.0	13,9	12.6	17	24.0	23.8
			T		13.8	12,9	13.2	14. 1	22.9	26.7
	3920795	VAR-C-DOX	L		25,4	32.4	38,9	35.1	42.0	43.5
			T		21.1	22.7	26.9	30.0	33.7	34.1
3.0	E5565	AM-SI/AI-DOX	L			9.5	9.4	11,2	13.2	18.6
			T			8.5	8.3	9.3	15,0	17.8
	3920835	Si/Al-DOX-VAR	L		14.1	12,0	10.0	13.0	19.5	29,1
			T		10.6	7.9	16.0	12.5	21.0	20,6
	3920795	VAR-C-DOX	L		<b>33.</b> 8	40.9	38.3	46.4	45, 8	46.7
			T		32.4	28.6	33, n	30,1	36.5	46.8
	•	Precracked	Charpy (PCC	) Impact Proper	ties (W/A	), in,-1	b/in. <sup>2</sup>			
0.750	E5565	AM-Si/A1-DOX	L	183.3	<u>:</u>		~~	560	880	1359
			T	182.9				512	701	1179
	3920835	Si/Al-DOX-VAR	L	184.2			779	1016	1405	
			T	185.5		~-	747	860	1483	
	3920795	VAR-C-DOX	Ĺ	182.1				2850	2811	
			T	181.8			1780	1912	2157	2273

#### Precracked Charpy Impact Properties

Precracked Charpy impact specimens of the type described earlier were used to evaluate specimens from the 0.75-inch plate of 9Ni-4Co (200) steels. The results given at the bottom of Table 45 can be compared with those for the maraging steels is ed in Table 38.

The performance of the precracked Charpy specimens confirmed the opinions about the effects of melting practice and anisotropy based on standard specimens. The ductile-brittle transition temperatures of the best heat, Heat No. 3920795, seemed to be below 0 F, and at or above room temperature for the other steels.

#### Environmental Effects

Fatigue-cracked tensile specimens of the type shown in Figure 36 were immersed in a 3 percent solution of NaCl in water at 70 F and subjected to a sustained load. Failure time for tensile loading, of various levels, for the sheet specimens are given in Table 46. The data can be compared with those in Table 39 for the 18Ni maraging steels.

TABLE 46. FAILURE TIMES FOR FATIGUE-PRECRACKED TENSILE SPECIMENS OF 9NI-4Co STEELS IMMERSED IN 3 PERCENT NACI SOLUTION AT 80 (4)

Heat Number Sheet Thickness)	Grade	Melting Practice	0.2 Percent Offset Yield Strength, itsi	Notch Tensile Suength, ksi	ercent of Notch Tensile Strength	Initial Applied Stress, ksi	Test Time, hr:min
3321246	250	AM-ISLVD	236.9	172.0	100		
(0.045 in.)				164.0	100		
					90	153,2	0;32
					90	150.4	0:37
					80	132.1	0:42
					80	135.7	0;38
					70	117.6	1:15
					70	118.6	1:10
3920851	250	Si/Al-DOX-VAR	233.6	163.9	100		
(0.045 in.)				178.5	100		
• • • •					90	153.3	0
					90	154.0	0
					80	137.0	0
					80	133.8	0:19
					70	119.0	0:39
					70	121.0	0:17
3920852	250	VAR-C-DOX	286.9	178.2	100		
(0.045 in.)			•	183.2	100		
					90	164.4	1:06
					90	160.4	1:08
			•		80	143.4	1:04
					80	143.7	1:33
		•		•	70	127.8	1:32
					70	127.2	1:54
3321246 (0.160 in.)	250	AM-D	238,4	66.4 64.2	100		
				75.7 <b>6</b> 7.8	100		
				91.0	80	59.5	1;30
					80	58.9	0:38
			_		70	50.7	2:57
					70	55.2	1:17
3920851	250	Si/Al-DOX-VAR	240.6	77.3	100	~*	
(0.160 in.)	200	Olyni-box via	240,0	76.8	100		
(0. 100 111.)				70,0	90	70.9	0:29
			•		90	69.3	1:12
		•			80	59.7	2:14
					80	63.7	0:50
					70	55.8	1:56
					70	51.5	1:19
3920852	250	VAR-C-DOX	234.3	73.6	100	••	
(0.160 in.)	200	0 500	<b>,-</b>	74.6	100		
far von mit				- <del>- • •</del>	90	68.9	
					90	68.5	0:14
					80	60.5	0:07
		•			80	62.5	0:22
					70	53,8	1:40
					70	54.2	1:23

<sup>(</sup>a) Longitudinal specimens of the shape shown in Figure 36.

The time for brittle fracture under sustained loading was essentially independent of the melting practice and sheet thickness of the 9Ni-4Co steels. As is usually the case, failure time increased as the loading stress decreased. However, all of the specimens broke in less than 3 hours. The lives were very short compared to those for the maraging specimens which withstood loads of 70 percent of the yield strength for 24 to 57 hours. Measurements on fractured surfaces indicate that the rate of crack growth were 4000  $\mu$ in. /min for the 9Ni-4Co sheet and 200  $\mu$ in. /min for the maraging steel with equivalent yield and notch-tensile strengths.

# Fracture Toughness

The types of specimens used to evaluate the fracture toughness of the (250) sheet and (200) plate of 9Ni-4Co steel are shown in Figures 37, 38, 39, and 40. The procedures and calculations were described in connection with the discussion on flat-rolled maraging steel products.

The results of the center-notched tensile tests on the 9Ni-4Co (250) sheet specimens are given in Table 47. Some of the tests were invalid by ASTM definitions because the net stress exceeded 80 percent of the yield strength. They show, however, that the air-melted steel had poorer plane-stress fracture toughness than the other two materials when tested as 0.045-inch sheet. Although thicker sheet did not show a similar trend, vacuum-arc remelting is considered beneficial. Specimen orientation did not appear to affect the  $K_{\rm C}$  values significantly.

TABLE 47. FRACTURE TOUGHNESS PROPERTIES OF HEAT TREATED 9NI-4Co (250) SHEET (4)

Thickness,			0.20 Percent Offset Yield Strength, ksi		K <sub>C</sub> V	alue, /in.
inch	Heat Number	Melting Practice	L	T	L	T
0,045	3321246	AM-D	236.9	232.0	157	142
	3920851	SI/AI-DOX, VAR	<b>23</b> 3.6	235.1	173	157
	3920852	VAR-C-DOX	227.6	236,9	161	171
0. 160	<b>33</b> 2 1246	AM-D	238.4	238.5	67	68
	3920851	SI/A1-DOX, VAR	240.6	240.8	65	68
	392 0852	VAR-C-DOX	234.8	237.8	71	68

<sup>(</sup>a) Determined in specimens of the type shown in Figure 22.

Table 48 summarizes the results of plane-strain fracture-toughness tests on flat tensile specimens from 0.160- and 0.375-inch stock. The  $K_{IC}$  values of the longitudinal specimens of 9Ni-4Co (250) sheet steel were low, ranging from 40 to 49 ksi  $\sqrt{in}$ , less than half of those for the maraging steels of the same gage and approximately equal strength. The differences in melting practices had no significant effect on the value. The plane-strain fracture toughness of the 3/8 and 3.0-inch plates did not vary significantly with direction of testing. The  $K_{IC}$  values of the VAR-C-DOX type of steel were higher than those for materials made by the two other melting practices. The difference was more pronounced for the thicker plate. The data also indicate that toughness decreased as plate thickness increased.

TABLE 48. FRACTURE-TOUGHNESS VALUES, K<sub>IC</sub>, FOR LONGITUDINAL AND TRANSVERSE SPECIMENS OF 9Ni-4Co STEEL<sup>(a)</sup>

Sheet Thickness,				Yield S	ent Offset trength, si		/aluc, /in.
inch	Heat Number	Grade	Melting Practice	L	T	L	T
0.160	3321246	250	AM-D		238.5		43
0.160	3920851	250	Si/Al-DOX, VAR		240.8	~ ■	43-1/2
0.160	3920852	250	VAR-C-DOX		237.8		47
0.375	<b>£55</b> 65	200	AM-Si/A1-DOX	184,0	<b>183.</b> 0	113 120 <sup>(b)</sup>	105 115(b)
0.375	3920835	200	Si/Al-DOX-VAR	185.0	183.0	121 130 <sup>(b)</sup>	116 120 <b>(b)</b>
0.375	3920795	200	VAR-C-DOX	184,0	182.0	135 141 <sup>(b)</sup>	133 142 <sup>(b)</sup>
3.0	E5565	200	AM-Si/Al-DOX	184.0	183.0	<b>6</b> 6	66
3.0	3920835	200	Si/A1-DOX-VAR	184.0	184.0	97	91
3.0	3920795	200	VAR-C-DOX	180.0	180.0	145	137

<sup>(</sup>a) The values were determined on specimens of the type shown in Figures 37, 38, and 40. The samples from the 3-inch plate were tested by Boeing Company, Seattle.

Difficulties were encountered in fatigue cracking specimens, of the type shown in Figure 39, from the 3/4-inch plate of 9Ni-4Co (200). It appeared that this (200) steel had an unusually high resistance to crack growth in bending fatigue. Because of the trouble in preparing specimens, no valid  $K_{\rm IC}$  value were obtained on this plate product.

### Metallographic Examinations

Metallographic studies were made on samples from the 3/8, 3/4, and 3.0-inch plates of 9Ni-4Co (200) steel, Heat Nos. E5565, 3920795, and 3920835 (which represent different melting practices). All of the 9Ni-4Co steel products, except the 3-inch plate from the air-melted heat (Heat No. E5565), met the requirements of cleanliness specification RSC-AF-2A established for this program. That particular plate product contained higher-than-acceptable amounts of oxide inclusions. The grain size of all materials appeared to be ASTM 8 or finer although difficulty was experienced in delineating the prior austenite grain boundaries. All samples had microstructures, typical for the material and heat treatment, which did not vary with specimen orientation. Although the microstructures of all products were quite similar, banding was slightly more severe in the 3/4-inch plate of the air-melted heat. For the 3-inch plate, banding was least pronounced in samples from Heat No. 3920835 (Si/Al-DOX-VAR).

<sup>(</sup>b) These  $K_{IC}$  values were calculated by the Paris equation instead of the Irwin equation given in the text and used for all other values in this report.

#### WELDING EVALUATION

# Objectives

An evaluation of the weldability of 18Ni maraging and 9Ni-4Co quenched and tempered steels was conducted by North American Aviation, Incorporation, Los Angeles Division, under subcontract to Republic Steel Corporation. The investigation included:

- (1) Determining the thermal cycles produced in the heat-affected zone (HAZ) by tungsten inert gas (TIG) welding procedures.
- (2) Determining the susceptibility of the steels to HAZ cracking as evidenced by loss of ductility on heating or during cooling.
- (3) Evaluating the mechanical properties of steel specimens that had been subjected to various thermal cycles which simulated the thermal cycles of welds.
- (4) Determining the mechanical properties of specimens welded by standard TIG procedures.

# Materials

In each phase of the program, both the 18Ni and the 9Ni-4Co alloy systems were evaluated in several thicknesses and compositions. The identity of the specific materials used in both phases was as follows:

Alloy	Heat No.	Thickness, inch
18Ni(250)VAR	3920864	0.160
	3920864	0.375
	3920864	0.750
18Ni(250)AM	53088	0.750
18Ni(300)VAR	08749	0.160
9Ni-4Co-0.45C VAR-C-DOX	3920852	0.160
9Ni-4Co-0.45C Si/Al-DOX-VAR	3920851	0.160
9Ni-4Co-0.25C VAR-C-DOX	3920795	0.375
,	3920795	0.750
9Ni-4Co-0.25C Si/Al-DOX-VAR	3920835	0.375
. ,	3920835	0.750

The chemical compositions of these heats may be found in Table 2 of this report. Weld wires used in the evaluations were of the following compositions:

Weld Wire for 18Ni Steels Weld Wire for 9Ni-4Co Steel

	TOINT DIEETS	7141-4C0 Stre1				
Elements	RSM 300 Weld Wire, percent	9Ni-4Co-0.25C, percent	9Ni-4Co-0.35C, percent			
C	0.025	0.25	0. 365			
Mn	0.08	0, 45	0.24			
Si	0.05	0.27	0.25			
P	0.011	0.005	0, 003			
S	0.007	0.004	0.004			
Ni	19.01	8.76	9. <b>4</b> 6			
Co	9. <b>04</b>	4, 57	3.92			
Mo	5.23	0.59	0.40			
Cr		0. <del>4</del> 8	0.32			
v	<b></b>	0.08	0.14			
Al	0.10					
Ti	0.72					

The HAZ test blanks and weld-test plates were rough cut from the sheet and plate stock so that the longitudinal axis of all test specimens would be perpendicular to the principal rolling direction. Each blank or test specimen was marked with an appropriate code designating its location with respect to the original sheet or plate and the material type.

The basic heat-treat cycle for all of the 18Ni material was as follows:

Solution anneal at 1500 F for 1 hr, air cool Re-solution anneal at 1500 F for 1 hr, air cool Age at 900 F for 3 hr, air cool.

The basic heat-treat cycle for the two grades of 9Ni-4Co material differed somewhat as follows:

# 9Ni-4Co(250) steel:

Austenitize at 1475 F for 1 hr, oil quench Refrigerate at -100 F for 2 hr Double temper at 450 F for 2 hr each, air cool.

# 9Ni-4Co(200) steel:

Normalize at 1650 F for 1 hr, oil quench Austenitize at 1550 F for 1 hr, oil quench Double temper at 1000 F for 2 hr each, air cool.

#### Simulated Heat-Affected Zone Studies

As a first step, the thermal cycle experienced by test plates 0.160 and 0.750-inch thick when welded by the TIG process was determined.

Chromel-alumel thermocouples 0.013 inch in diameter were welded into small holes drilled into the back side of the test plates. The test plate was then TIG welded while the thermocouple output was recorded on a Honeywell Visicorder. These data were then used in programming a Gleeble Model 510 thermal-cycle simulating machine. The Gleeble specimens were 0.50-inch wide if cut from 0.160-inch sheet, and either 0.375-inch in diameter or 0.420-inch square if cut from 0.750-inch plate.

# Hot Ductility Tests

Specimens were heated to a maximum temperature of 2600 F and broken in tension while being heated and while being cooled. The nil-ductility temperature, defined as the minimum temperature at which zero reduction of area was encountered on heating, was:

Heat No.	Grade	Nil-Ductility Temperature
11eat 140.	Grade	Temperature
08749	18Ni(300)VAR	2518
3920864	18Ni(250)VAR	2483
53088	18Ni(250)AM	2460
3920852	9Ni-4Co(250)VAR	2490
3920851	9Ni-4Co(250)Si/Ai-DOX-VAR	2567
3920795	9Ni-4Co(200)VAR	2590
3920835	9Ni-4Co(200)Si/Al-DOX-VAR	2580

The "on-heating" tensile tests were started at approximately 1200 F and made at increments of 200 to 300 F until the nil-ductility temperature was approached, where a number of duplicate tests were made. Both temperature and load were determined for each specimen. All of the steels exhibited large reductions of area until just before the nil-ductility temperature was reached and then dropped precipitously. All the heats may be considered to be more or less "normal" in their "on-heating" behavior. On cooling, however, a noteworthy difference was observed among the 18Ni steels. The two 18Ni VAR heats recovered almost completely in ductility by the time the specimens had cooled to 2300 F. The air-melted heat exhibited erratic ductility to as low as 2000 F, and even at lower temperatures its ductility was less than the vacuum arcremelted heats. All of the 9Ni-4Co heats recovered ductility very quickly after cooling was started but lost ductility on cooling to 1800 F and lower. The loss in "on cooling" ductility below 1800 F was thought to be associated with the grain coarsening which these steels experienced when heated to 2500 to 2600 F.

# Room-Temperature Tests on Simulated HAZ Specimens

The mechanical properties of the various regions in the HAZ were determined on material that had been subjected to weld simulating thermal cycles in the Gleeble equipment using various peak temperatures. No load was applied during the thermal

cycle. The specimens were then machined into tensile and Charpy V-notch specimens. Because the decomposition products formed in the HAZ of most high strength steels are significantly harder than the base material, the tensile bars were mildly notched in the test section prior to testing. The results of the tensile and impact tests were used to evaluate the strength and toughness of the metallurgical structures produced at each of the peak temperatures. Also evaluated were the effects of pre- or post-weld heat treatment.

18Ni Steels. Notched tensile-strength specimens were prepared from the 0.160-inch sheet of 300 VAR steel, Heat No. 08749, 0.750-inch plate of 250 VAR steel, Heat No. 3920864, and 0.750-inch plate of 250 AM steel, Heat No. 53088. Each of these steels was solution annealed at 1500 F for 1 hour and air cooled, then aged at 900 F for 3 hours and air cooled prior to being placed in the Gleeble equipment. Tested without any post-weld simulating aging treatment, the notch tensile strength of 0.160-inch sheet, 18Ni(300) VAR steel, dropped 20 percent below that of the parent metal value when cycled at a peak temperature of 1200 F. It dropped further to about 40 percent when cycled at temperatures of 1800 and 2530 F. This drop in strength at 1200 F was attributed to austenite reversion and overaging while that at 1800 and 2530 F was attributed to complete re-solution. Similar specimens of these three steels were tested after reaging for 3 hours at 850, 900, and 950 F. Reaging (after the weld simulating thermal cycle) restored the strength of the specimens to that of the parent metal for all practical purposes.

The effect of peak temperature cycles on 0.750-inch plate, 18Ni(250) VAR steel, was similar to that described for the 18Ni(300) grade material. The notch tensile strength decreased by about 20 percent from the value for the parent metal after cycling to a peak temperature of 1200 F and by about 40 percent after cycling to 1800 and 2500 F in the absence of reaging. Tests to determine the effect of reaging temperature were conducted on specimens cycled through 1200, 1800, 2400, and 2500 F. Again, it was found that reaging after the weld simulating thermal cycle restored the strength of the material to that of the "parent metal".

Additional specimens of the 18Ni(250) VAR steel (Heat No. 3920864) were cycled over the range from 1000 to 1500 F and aged for 3 hours at 900 F before testing. After cycling at a peak temperature of 1300 F, the strength was 5 percent below that of the parent steel, indicating that a loss in strength of about 5 percent can be anticipated in the HAZ after single pass welding. Tests were also conducted on the steel by simulating conditions which would exist during multipass welding. Specimens were subjected to ten thermal cycles starting at 1400 F and decreasing in peak temperature by 50 F per cycle to 950 F. As cycled, the strength was reduced about 5 percent below that previously obtained after a single cycle at 1200 F. Reaging the material subjected to ten thermal cycles at 850, 900, and 950 F increased the strength only 5 to 10 percent. This increase compared with 20 to 25 percent increase in strength obtained upon reaging specimens subjected to a single cycle at a peak temperature of 1200 F. Simulated multiple weld thermal cycles in the 950 to 1400 F temperature range, therefore, resulted in sufficient overaging of the martensite as well as in austenite reversion to prevent full strengthening at the reaging temperatures.

The 0.750-inch plate of 18Ni(250) AM steel was tested for notch tensile strength after cycle heating and reaging at 900 F for 3 hours. As cycled, the strength decreased with increasing peak temperature in much the same way as did the VAR grades of steel.

Reaging at 900 F increased the notch tensile strength to 95 percent of that of the parent steel after all peak cycle temperatures up to 2420 F. As the peak cycle temperature was increased up to and within the nil ductility range of 2450 to 2500 F, the reaged notch tensile strength decreased rapidly. This behavior was in contrast to that for the two vacuum arc remelted steels where strength could be restored for all practical purposes even after heating to 2500 F.

The effect of simulated weld thermal cycles on fracture toughness of the HAZ was determined for 0.750-inch plate of the 18Ni(250) AM and VAR steels, Heat No. 53088 and Heat No. 3920864, by employing impact blanks from which precracked Charpy (PCC) specimens were machined after cycling. These were standard-size Charpy V-notch specimens that contained, at the base of the V-notches, fatigue cracks from 0.015 to 0.037-inch deep formed by cyclic reverse bending. All specimens of the 18Ni(250) VAR steel, which were cycled through peak temperatures of 1200 F and 1800 F and subsequently reaged at 850, 900, and 950 F, exhibited fracture-toughness values equivalent to or greater than the parent material that had been aged at 900 F for 3 hours. Fracture-toughness values were expressed in terms of W/A, where W is the impact energy in inch-pounds (in.-1b) and A is the fracture area in square inches (in.2). Cycling through 2500 F and reaging reduced the W/A values below the parent material range, i.e., to values between 460 and 830 in.lb/in.2. The values for the parent material were about 1000 in.-1b/in.2.

The 18Ni(250) AM steel had a high incidence of delamination in the fractured surfaces, which resulted in considerable scatter and generally higher PCC fracture-toughness values than were obtained for the VAR steel. Because the spread in fracture-toughness values for the air-melted steel cycled to any peak temperature was so large, no meaningful conclusions could be drawn as to the effect of peak temperature.

#### 9Ni-4Co Steels

The effect of weld thermal cycles on notch tensile strength was determined on two 9Ni-4Co(250) VAR steels (VAR-C-DOX steel, Heat No. 3920852, and Si/Al-DOX-VAR steel, Heat No. 3920851) and two 9Ni-4Co(200) VAR steels (VAR-C-DOX steel, Heat No. 392079, and Si/Al-DOX-VAR steel, Heat No. 3920835). The 9Ni-4Co(250) steels were tested as 0.160-inch sheet, while the 9Ni-4Co(200) steels were tested as 0.750-inch plate.

The two 9Ni-4Co(250) steels in the form of 0.160-inch sheet were subjected to thermal cycling to various peak temperatures in the annealed condition. Subsequently, they were austenitized at 1475 F for 1 hour, oil quenched, refrigerated to -100 F for 2 hours, warmed in air, then double tempered at 450 F for 2 hours each and air cooled. To investigate the effect of tempering temperature, some of these specimens were then double tempered again at 450, 500, and 550 F prior to testing. No significant effect of peak temperature on notch tensile strength was observed for either the carbon or silicon deoxidized grade for cycles in the range of 1400 to 1800 F. At both of these peak temperatures, the effect of final tempering temperature was similar. Highest notch tensile values were obtained upon tempering at 450 F, the values approximating those for the parent steel. Notch tensile values after tempering at 500 and 550 F were almost equivalent but slightly lower than for steel tempered at 450 F. Specimens cycled through 2580 F exhibited considerable scatter and a number of low values. This scatter was evident in steels deoxidized by either technique and after tempering at all

three tempering temperatures. It was attributed to a transition from transgranular to grain-boundary failure coupled with delamination, which predominated in this specimen group.

The 9Ni-4Co(200) steels were tosted at 0.750-inch plate and were thermal cycled in the fully heat-treated condition. The plate had been normalized at 1650 F for 1 hour and air cooled, austenitized at 1550 F for 1 hour, oil quenched, and double tempered for 2 hours each at 950, 1000, or 1050 F prior to thermal cycling at various peak temperatures. Duplicate specimens were tested either as cycled or as stress relieved at 50 F below the initial tempering temperature. The notch tensile strength for the 9Ni-4Co(200) steel deoxidized by either of the two techniques, increased rapidly with increased peak temperature in the range of 1300 to 1500 F when the specimens were tested in the as-cycled condition. This rapid increase in strength could be accounted for by the greater percentage of material transforming to austenite during heating and to fresh martensite on cooling. As the peak temperature was increased beyond the range of 1500 to 1600 F, the notch tensile strength of the steel in the as-cycled condition decreased slightly. With a peak temperature of 2600 F, it dropped rapidly and became erratic. Fractures of specimens cycled through peak temperatures up to 1800 F without stress relieving tended to be of the cleavage type. As the peak cycle temperature was increased to 2500 F, fractures became partially intergranular. Gross intergranular fractures and large splits occurred upon increasing the peak cycle temperature to 2600 F.

Stress relieving after thermal cycling at 50 F below the initial tempering temperature had a pronounced effect on the notch tensile strength of the steel. Such stress relieving completely eliminated the increased strength obtained by transformation from austenite to martensite on cooling that had been observed after peak cycling above 1300 F. For both deoxidation techniques, the steels in the stress-relieved condition, after thermal cycling at temperatures up to 2500 F, had notch tensile strengths that were equivalent to those of the base steels. It was obvious that the stress-relief treatment tempered the fresh martensite formed during the thermal cycling.

Studies were undertaken to determine the effect of simulated weld thermal cycles on HAZ fracture toughness of the 9Ni-4Co(200) steel produced by both the carbon and silicon-aluminum deoxidation techniques. The materials used in these studies comprised PCC blanks from 0.750-inch-thick plate of grades VAR-C-DOX, Heat No. 3920795, and Si/Al-DOX-VAR, Heat No. 3920835. The steels were tempered at 950, 1000 or 1050 F, then cycled, and, where applicable, stress relieved at 50 F below the original tempering temperature. The fracture toughness of the VAR-C-DOX grade parent-metal material was quite high in all its original tempers, the toughness values being on the order of 2500 to 3200 in. -lb/in. 2. The values were somewhat higher when tempered at the higher temperatures.

Thermal cycling at peak temperatures of 1400 and 1800 F decreased the fracture toughness (W/A value) of the simulated HAZ to about 40 percent of that of the base steel. The effect of prior tempering temperature was still apparent after cycling to 1400 F. \*\* was eliminated, however, after cycling through 1800 F. The fracture toughness after cycling to 1400 and 1800 F was raised by subsequent stress relieving, reaching values equivalent to about 70 percent of those of the base steel. The beneficial influence of originally tempering at 1050 F prior to cycling was apparent from the fact that after thermal cycling to 1400 and 1800 F and stress relieving at 1000 F, toughness values were the same. Test results showed considerable scatter and decreased toughness after cycling through 2600 F. The mode of failure after cycling through this temperature was intergranular.

The Si/Al-DOX-VAR steel was much lower in fracture toughness than the carbon deoxidized teel. The difference was as much as 40 to 60 percent. Fracture toughness without str ss relief after cycling either to 1400 or 1800 F decreased as much as 70 to 80 percent from that of the base steel, irrespective of the prior tempering temperature. Toughness after cycling to 1400 F was less than after cycling to 1800 F. The effect of prior tempering temperature was still evident in steel cycled to 1400 F. Stress relieving of the steel after cycling had a marked effect on its fracture toughness. Specimens cycled through 1400 and 1800 F and stress relieved at 900 F showed fracture-toughness values that were about equivalent to those of the parent steel. Stress-relief treatments at 950 and 1000 F were less effective. These treatments raised the fricture-toughness values only to about 70 percent of values for the parent steel. Fractured surfaces of specimens cycled at 2600 F showed grain-boundary failure, splitting, and some oxidation along the split surfaces.

The results of the PCC fracture-toughness experiments showed the toughness of the 0.25 percent carbon 9Ni-4Co steel made by the VAR-C-DOX melting technique to be much superior to that of steel made by the Si/Al-DOX-VAR melting technique.

# Transweld Properties of TIG Weldments

Transweld properties of weldments made by the TIG (tungsten-arc, inert-gas) process were determined for both the 18Ni and the 9Ni-4Co steels using specimen thicknesses of 0.160, 0.325, and 0.750 inch. Weld test specimens were prepared by joining plate or sheet 14 to 18 inches long. A summary of the materials, thicknesses, and heat-treat sequences used was as follows:

Material	Heat No.	Thickness,	Heat Treat-Weld Sequence
18Ni(250) VAR	3920864	0.160	Anneal. Solution treat
18Ni(300) VAR	08749	0.160	1500 F/1 hr and air cool.
18Ni(250) VAR	3920864	0.375	Weld using RSM-300 welding
18Ni(250) VAR	3920864	0.750	wire. Age 900 F/3 hr. Test.
9Ni-4Co(250) VAR-C-DOX	3920852	0.160	Anneal. Weld using 9Ni-4Co-
9Ni-4Co(250) Si/Al-DOX-VAR	3920851	0.160	0.35C wire. Austenitize 1475 F/1 hr and oil quench. Refrigerate -100 F/2 hr. Double temper 450 F/2 hr each. Test.
9Ni-4Co(200) VAR-C-DOX	3920795 3920795	0.375 0.750	Anneal. Normalize 1650 F/
9N; -4Ca!200\ S; /A1 -DOY - VAP	3920835	0.375	Austenitize 1550 F/1 hr and
9Ni-4Co(200) Si/Al-DOX-VAR	3920835	0.750	oil quench. Double temper 1000 F/2 hr each. Weld using 9Ni-4Co-0.25C wire.

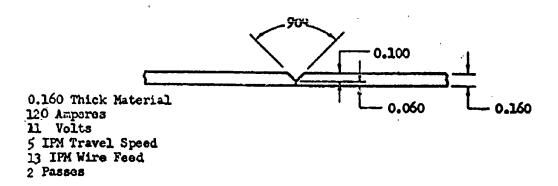
All test specimens were made with the welding direction parallel to the principal rolling direction of the sheet or plate. None of the specimens was stress relieved and no preheat or postheat was employed. All steels used were made by the vacuum-arcremelt technique. After welding, the test materials were inspected using standard radiographic and dye-penetrant techniques. Transweld-bend, tensile, partialthickness-crack (PTC) fracture-toughness, and metallographic specimens were prepared. Specimens were evaluated using tests on bend, chemical analysis, tensile with weld bead left on, and PTC fracture toughness with weld bead removed. Weld variables and energy inputs were maintained consistent with those used in developing the thermalcycle data for the simulated HAZ tests, where the energy inputs were in the range of 15,000 to 20,000 joules/in. for the 0.160-inch-thick material and 22,500 to 38,000 joules/in. for the 0.750-inch-thick material. The machine settings and welding parameters for each group of test plates and the weld-joint designs were as shown in Figure 41. The test-specimen configurations for the face and side bend, the transweld tensile, and the partial-thickness-crack (PTC) fracture-toughness tests were as shown in Figures 42 and 43. All static transweld tensile tests were made with the weld bead left on. Specimens were tested at strain rates of 0.03 to 0.05 in./in./min with either a 1-inch or 2-inch averaging extensometer. Elongation was measured on all specimens, but reduction of area was measured on the round bar specimens only.

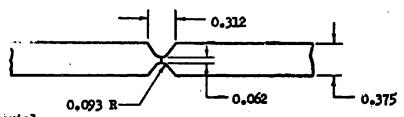
#### 18Ni Steels

The 18Ni steels were welded in the resolution-treated condition using RSM 300 grade vacuum-melted wire and aged for 3 hours at 900 F after welding. The chemical composition of the wire was recorded earlier in the welding-evaluation section under "Materials". Tests were conducted on 18Ni(250) VAR steel in 0.160-inch, 0.375-inch, and 0.750-inch thicknesses, and on 18Ni(300) VAR steel in 0.160-inch thickness. In the 0.160-inch material, weld tensile strength was equivalent to that of the parent material, as failures occurred outside the weld zone. In the 0.375-inch and 0.750-inch thicknesses, failures occurred in the weld metal at yield and ultimate-tensile-strength values equivalent to 90 to 100 percent of those of the parent steel. Ductility was only one-third that of the parent steel, however. The significant decrease in ductility was attributed to the coarse-grained dendritic structure constituting the cast fusion zone. Tables 49 and 50 cite the tensile properties of the parent metal and the tensile properties after welding.

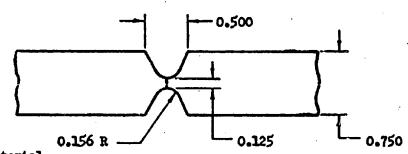
A number of bend tests were made using a guided bend test fixture and the procedure specified by MIL-S-418. Face bends were made on the 0.160- and 0.375-inch material, while side bends were made on the 0.750-inch material. A 2-T bend radius and a head travel rate of 1 in./min were maintained for all tests. Failing load and bend angles were recorded for each specimen. Results are summarized in Tables 51, 52, and 53. All failures occurred in the fusion zone. Face bend ductility for the 18Ni(250) and (300) steel in thickness of 0.160 inch was almost equivalent. Side bend testing of material 0.750-inch thick showed generally good ductility, except where there was some lack of side-wall fusion. In the latter case, the bend ductility was about 25 percent of that for specimens that were free of defects.

Fracture-toughness testing of transweld tensile specimens was conducted using the partial-thickness-crack (PTC) method. Crack starters were prepared by electrical-discharge machining a groove in the HAZ parallel to the weld direction. The tests were conducted with the weld bead ground flush and with the groove at the fusion line. Semielliptical cracks were grown by bending fatigue at loads between 30 and





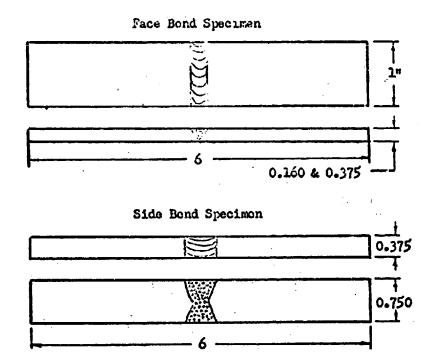
0.375 Thick Material 175 Amperes 11 Volts 5 IPM Travel Speed 15 IPM Wire Feed 7 Passes



0.750 Thick Material 200 Amperes 12.5 Volts 4 IPM Travel Speed 29 IPM Wire Feed 15 Passes

# FIGURE 41. WELD-JOINT DESIGNS AND WELDING PARAMETERS

Dimensions in Inches.



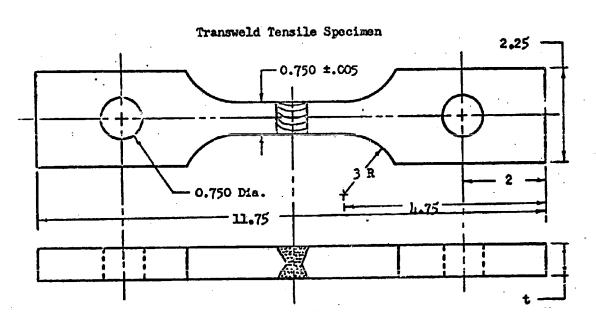


FIGURE 42. FACE- AND SIDE-BEND AND TRANSWELD-TENSILE-SPECIMEN CONFIGURATIONS

Dimensions in Inches.

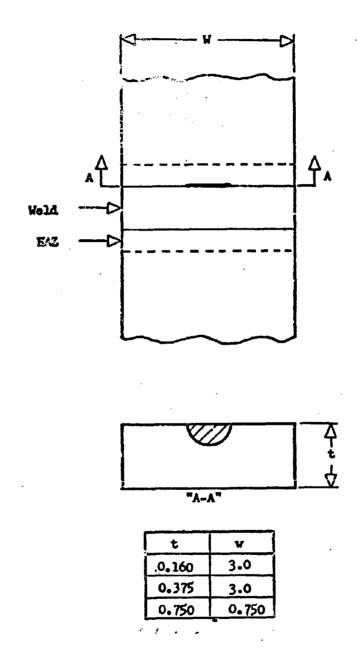


FIGURE 43. PARTIAL THICKNESS FRACTURE TOUGHNESS SPECIMEN
Dimensions in Inches.

TABLE 49. TENSILE TESTS ON PARENT-METAL SPECIMENS

	Heat Treat	Specimen	Thickness,	F <sub>tu</sub> ,	Fry,	Percent E	longation	Reduction of Area
Material <sup>(a)</sup> Con	Condition	Number is	inch		F <sub>ty</sub> , ksi	2-in, G.L.	1-in, G.L.	percent
18Ni (250)	(1)	864-1	0.168	256,9	247.7	9		
	(1)	864-2	0.168	257.2	244,8	9		
18N1 (250)	(1)	8-15	3/4	265.0	257.5		11.0	56
	(1)	U-20	3/4	264.0	256,8		12.5	56

<sup>(</sup>a) Properties of the 18Ni(300) grade steel were not listed.

TABLE 50. TENSILE RESULTS OF WELDED SPECIMENS

Material	Heat-Treat Condition	Specimen Number	Thickness, inch	F <sub>tu</sub> . ksi	F <sub>ty</sub> . ksi	Percent Elongation in 2 Inches	Failure Location(a)
18Ni	(1)	18Ni36	0. 169	258.7	249, 9	7	PM
RSM 300	(1)	18Ni37	0.168	256.4	249.9	. 8	PM
	(1)	18Ni38	0. 169	256.1	248.9	8	PM
18Ni	(1)	18Ni22	0, 164	274.4	271.9	8	PM
RSM 300	(1)	18Ni23	0.163	274.0	267.9	8	PM
	(1)	18Ni24	0. 163	275, 0	269, 1	10	PM
16Ni	·(1)	18Ni10	0.395	263.3	260.2	4	WM
RSM 250	(1)	18Ni11	0.40	265.3	259.6	3	WM .
18Ní	(2)	18Ni2	0.783	250.8	237.4	3	WM
RSM 250	(2)	18Ni4	0.783	254,4	244.0	4 .	WM

# Heat Treatment:

<sup>(1)</sup> Anneal, weld, austenitize 1475 F/1 hr, oil quench; refrigerate -100 F/2 hr, air warm; double temper 450 F/2 hr each, air cool.

<sup>(2)</sup> Anneal; weld; age 900 F/3 hr; air cool.

<sup>(3)</sup> Normalize 1650 F/1 hr; air cool; austenitize 1550 F/1 hr; oil quench; double temper 1000 F/2 hr each; sir cool; weld.

<sup>(</sup>a) PM = parent metal; WM = weld metal.

TABLE 51. GUIDED-FACE BEND-TEST RESULTS FOR 0. 160-INCH-THICK MATERIAL

Materi <b>al</b>	Specimen Number	Width, inch	Thickness,	Maximum Bending Load, 1b/in, <sup>2</sup>	Bend Angle, deg	Failure Location <sup>(a)</sup>
18Ni (250)	18Ni40	1.000	0.149	\$060	27	₩NI
	18Ni41	0.998	0 <b>.</b> 149	3050	17	₩Ni
	18Ni42	0.996	0.149	3020	17	WM
	18Ni43	0.998	0.149	3060	21	WM
	18Ni44	0.999	0.149	2960	18	WM
	18Ni45	0.996	0.148	2970	17	WM
	18Ni46	0.998	0.148	2980	17	WM
	18Ni <b>4</b> 7	0.999	0.149	3180	40	WM
18Ni (300)	18Ni25	1.000	0, 155	3180	13	WM
	18Ni26	1.000	0.155	3370	19	WM
	18Ni27	1.000	0.155	3300	15	₩M
	18Ni28	1.000	0,155	3320	18	, WM
	18Ni29	1.000	9.150	3270	15	WM
	18Ni30	1.000	0.155	- 3400	18	WM
	18Ni31	1.000	0.155	3360	17	WM
	18Ni32	1.000	<b>0.</b> 155	3380	23	₩M

<sup>(</sup>a) WM = weld metal.

TABLE 52. GUIDED-FACE BEND-TEST RESULTS FOR 0, 375 IN. THICK MATERIAL

Material	Specimen Number	Width, inch	Thickness, inch	Bending Load, 1b/in, 2	Bend Angle, deg	Failure Location
18Ni (250)	18Ni15	1.025	0.363	11,250	10	<b>W</b> M
• •	18Ni16	1,026	0.356	11,000	16	₩M
.*	18Ni17	•1. <b>02</b> 6	0.363	11,500	15	₩M
	18Ni18	1,026	0,363	11,500	14	<b>W</b> M

TABLE 53, GUIDED SIDE BEND TEST RESULTS FOR 0.750-INCH-THICK MATERIAL

	· Maximum										
	Specimen Number	Specimen Width.		Bending Load	Bend	Failure					
Material		inch	inch	1b/in, <sup>2</sup>	Angle, deg	Location					
18Ni (250)	18Ni6	0.726	0.372	8650	27	WM					
(,	18Ni7	0.726	0.371	7550	7	. WM(a)					
	18Ni8	0.725	0.372	8000	7	WM					
	18Ni9	0.726	0.373	9000	31	<b>W</b> M					

<sup>(</sup>a) Tear initiated at point where lack of sidewall fusion occurred.

45 percent of the yield strength of the parent steel and with an R factor of  $\pm 0.4$ . Cracks were grown to a geometry where it was calculated that the gross area stress would not exceed the yield stress and the crack depth would not exceed the half thickness of the material. The specimens were tested to failure using a load rate of 150,000 psi/min. Efforts were made to measure "pop-in" with a separable averaging extensometer. Pop-in is the load at which initial crack instability occurs and is the load used in calculating the plane-strain critical stress-intensity factor,  $K_{\rm IC}$ . Data are shown in Table 54. The high stress at fracture casts doubt on the validity of the tests in evaluating fracture toughness. Soft areas were found to exist in the HAZ, and these apparently affected the advancing crack front.

Macrosections and hardness surveys were made for each material thickness and grade in the as-welded and the welded and aged conditions. In the as-welded condition, the Vickers hardness in the center section of the weld area increased with increasing plate thickness. When aged after multipass welding, however, the hardness in the center section of the plate decreased with increasing plate thickness. These effects resulted from aging of the martensite and austenite reversion. The quantity of reverted austenite increased to the point where considerable softening in the center of the weld zone occurred in the 0.750-inch plate of 18Ni(250) steel. In the thinner 0.160-inch materials, the hardness values in the weld and parent metal after aging were essentially the same at all locations, reaching levels of approximately 580 VPM and 560 VPM, respectively, for the 18Ni(300) and the (250) grades of steel.

# 9Ni-4Co Steels

The 9Ni-4Co(250) steels (containing approximately 0.45 percent C) were fully heat treated after welding whereas the 9Ni-4Co(200) steels (containing approximately 0.25% C) were heat treated before welding and tested in the as-welded condition. The telling procedures were the same as those described for the 18Ni steels. Tables 55 and 56 compare the tensile properties of the parent rietal with those of welded specimens. It will be noted that the welded specimens were as strong, both in yield strength and tensile strength, as the parent metal. The ductility cannot be directly compared because the welds were not ground.

Face bends in 0.160-inch-(nick sheet of both grades of 9Ni-4Co(250) steel showed no significant difference in bend angle or failing load with difference in melting procedure. Four of the specimens from the VAR-C-DON material showed some lack of interpass fusion on the fracture surface. Two specimens showed some scattered porosity. Only one of the six specimens showed any significant decrease in bend angle, however. In both thicknesses, the bend ductility was slightly less for steel of the Si/Al-DOX-VAR grade than for the VAR-C-DOX grade. It is significant that of the specimens from 0.750-inch plate, all from the VAR-C-DOY steel and three of the four from the Si/Al-DOX-VAR steel were bent to a 160-degree angle without failure. Two specimens contained slight tears in the weld metal, but these did not propagate. The excellent weld ductility may, in part, be credited to the tempering that resulted from multiple weld passes in the thicker plate. Another factor that may have contributed to the apparent increase in ductility is the different direction of testing. (Side bends were used on tests of 0.750-inch plate, while face bends were used on the thinner material.) Results of the bend tests are shown in Tables 57, 58, and 59.

TABLE 54. TRANSWELD PTC FRACTURE-TOUGHNESS TEST RESULTS FOR 18NI STEEL  $^\circ$ 

	Thickness.	Crack Length.	Crack Depth,	Grack Area		<b>6</b> !	
Material	inch	inch	inch	1n. 2	Gross	Net	Kic
250 Grade VAR	0, 150	0, 220	0.063	0,0108	250,5	256.7	104.5
	0, 152	0.250	0.061	0.0119	252,9	259.6	.109.4
300 Grade VAR	0, 152	0.221	0.064	0.0111	257.6	264.0	107.2
	0, 153	0.236	0.066	0.0122	260.7	267.8	111.5
250 Grave VAR	0, 355	0. 555	0, 153	0.0666	216.6	231.1	139.2
	0, 344	0.378	0,103	0.0306	249.9	257.7	103.4
	0, 344	0.630	0.167	0.0826	204.6	222.4	103.4
250 Grade VAR	0.725	0.248	0.092	0.0179	240, 9	249.4	109.5
	0.726	0,390	0.147	0.0450	231,1	251.8	131.2
	0,725	O. 49Z	0.175	0. 6676	203.9	232.7	127.8

TABLE 55. TENSILE RESULTS OF PAKENT-METAL SPECIMENS

•	Heat Treat	Specimen	Thickness,	f <sub>m</sub> ,	F <sub>ty</sub>	Percent E	longation	Reduction of Area,
Material	Condition	Number	inch	ksi	ksi	2-in. G. L.	1-in. G.L.	percent
9N1-4Co (250)	(1)	£52-1	0.170	254, 1	218.1	7	**	
VAR-C-DOX	(1)	852-2	0, 161	264.7	224.1	8		••
9Ni-4Co (250)	(1)	<del>6</del> 51-1	0.170	273.0	231,6	7	<b></b> .	
Si/Al-DOX-VAR	(1)	651-2	0.168	273.8	232.4	7		
9Ni-4Co (200)	(2)	A-4	3/4	195.5	179.2		18, 5	60
VAR-C-DOX	(2)	D-25	3/4	198.2	182.2		17.5	57
	(3)	<b>B-11</b>	3/4	197.6		*-	18.0	59
	(3)	£-21	3/4	197.7	184.0		18.0	60
	(4)	C 16	3/4	192,6	181.0		18,5	60
	(4)	<b>F-8</b>	3/4	191.5	180.7	**	18, 5	63
9Ni-4Co (200)	(2)	J-1	3/4	209.0	i <b>94.</b> 0		18.0	55
Si/Ai-DOX-VAR	(2)	M-16	3/4	209.0	183.2		16, 5	54
	(3)	<b>K</b> -6	3/4	206.0	185.1	••	17.0	58
	(3)	N-21	3/4	202.6	186, 3	<b></b>	17.5	56
	(4)	L-11	3/4	201.0	184,0	••	18,0	58
	(4)	O-24	3/4	200, 1	183.6	-	18,5	57

### Heat Treatments:

- (1) Austenitize 147 F/1 hr, oil quench; refrigerate 100 F/2 hr, air warm; double temper 450 F/2 hr each, air cool.
- (2) Normalize 1650 F/1 hr air cool; austenitize 1550 F/1 hr, oil quench; double temper 950 F/2 hr each, air cool.
- (3) Same as (2), except temper at 1000 F.
- (4) Same as (2), except temper at 1050 F.

TABLE 56. TENSILE RESULTS OF WELDED SPECIMENS

Material	Heat Treat Condition	Specimen Number	Thickness, inch	F <sub>tu</sub> , ksi	F <sub>ty</sub> ,	Percent Elongation in 2 Inches	Failure Location
9Ni-4Co (250)	(1)	94A33	0.173	279.5	245.8	6	PM
VAR-C-DOX	(1)	94A34	0.174	279,9	241.3	. ,	₽M
	(1)	94A35	0, 175	279.6	242.7	Ą	PM
9Ni~4Co (250)	(1)	94B38	0.169	285,3	244.8	7	a M
Si/Al-DOX-VAR	(1)	94B34	0.169	284.1	244.8	€	PM
	(1)	<b>94B3</b> 5	0.169	283,7	242.9	7	₽M,
9Ni-4Co (200)	(2)	94A10	0.386	195.3	172.9	17	₽M
VAR-C-DOX	(2)	94A11	0.385	197.9	179, 2	16	<b>PM</b>
9Ni-4Co (200)	(2)	94B10	0.385	202.5	177.8	15	₽M
Si/Al-DOX-VAR	(2)	94b11	. 0.381	204,3	177.6	15	₽M
9Ni-4Co (200)	(2)	94A2	0.769	198.9	181.0	20	₽M
VAR-C-DOX	(2)	94 A 4	0.775	198.8	184.6	. 20	₽M
9Ni-4Co (200)	(2)	9482	0.767	209.5	185,1	· 18	₽M
SI/AI-DOX-VAR	(2)	94 <b>B</b> 4	0.770	209.9	183.5	20	PM

#### Heat Treatments:

<sup>(1)</sup> Anneal, weld, austenitize 1475 F/1 hr, oil quench; refrigerate -100 F/2 hr, air warm; double temper 450 F/2 hr each, air cool.

<sup>(2)</sup> Normalize 1650 F/1 hr, air cool; austenitize 1550 F/1 hr, oil quench; double temper 1960 F/2 hr each, air cool; weld.

<sup>(</sup>a) PM = Parent metal.

TABLE 57. GUIDED-FACE BEND-TEST RESULTS FOR 0, 160 INCH-THICK MATERIAL

Materiat	Specimen Number	Width, inch	Thickness,	Maximum Bending Load, 1b/in, <sup>2</sup>	Bend Angle, degrees	Failure	Location
11Ni-4Co (250)	94A3G	0 <b>. 99</b> 8	0, 159	3100	25	NIE(A)	₩₩ Þ)
VAR-C-DOX	94A37	0,998	0,154	3400	12	NIF	WM
	94 <b>A3</b> 8	0.997	0.154	2830	9	NIF	WM
	94A39	0.998	0.154	3420	30	NIF	WM
	94A40	0.990	0.149	3370	22	WM	
	94A41	1.002	0.164	3820	22	WM Por	rosity
	94A42	1,002	0.155	3250	16	WM Po	rosity
	94 <b>A4</b> 3	1.002	0.164	3980	25	WM	•
9Ni-4Co (250)	94B36	0,999	0, 143	3280	23	w	М
Si/Al-DOX-VAR	94B37	1,001	0,148	3540	21	W	M
•	94B38	0.994	0.138	3120	25	W	M
	94B39	1,004	0.135	3350	27	W	M
	94B40	0.999	0,138	3080	26	W	M
•	94841	1,001	0.1.3	3220	17	W	M
	94B42	0.999	0.147	3530	23	W	M
•	94643	1,009	0.142	3280	21	W	M

<sup>(</sup>a) NIF = No interpass fusion.

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TABLE 58, GUIDED-FACE BEND-TEST RESULTS FOR 0, 975-INCH-THICK MATERIAL

Material	Specimen Number	Width, inch	Thickness, inch	Maximum Bending Load, 1b/in. <sup>2</sup>	Bend Angle, degrees	Failure Location
9Ni-4Co (200)	94A15	1.005	0.347	9400	47	Weld Metal
VAR-C-DOX	94A16	1.009	0. 347	9450	47	Weld Metal
	94A17	1.005	0.342	. 9500	58	Fusion Line
	94A18	1,005	0.347	9750	48	Fusion Line
9N1-4Co (20°)	04815	1.012	0, 347	9400	28	Weld Metal
SI/AI-DOX-VAR	94216	1.009	0.347	9700	35	Weld Metal
	94B17	1,009	0.347	9800	42	Weld Metal
	94B18	1.010	0.347	9550	49	Weld Metal

TABLE 59. GUIDED-SIDE BEND-TEST RESULTS FOR 0, 750-INCH-THICK MATERIAL

Material	Maximum								
	Specimen Number	Width, inch	Thickness,	Bending Load, 1h/in. <sup>2</sup>	Bend Angle, degrees	Failure Location			
9N1-4Co (200)	94A6	0,726	0,372	10, 130	161	ido Failure(a)			
VAR-C-DOX	94A7	0,726	0.372	9,810	158	No Failure			
	94A8	0.726	0,373	10,140	161	No Failure			
	94 A9	0.796	0, 372	10, 130	160	No Failure (a)			
9NI-4Co (200)	<b>248</b> 6	0,726	0.372	10, 580	162	No Failure			
DI/AI DOX-VAR	94 B7	0.726	0,371	8,680	64	Weld Metal			
	8458	(.726	0,373	10,700	160	No Failure			
	9489	0.726	0,372	10,490	159	No Failure			

<sup>(</sup>a) Slight tears occurred on surface, did not propagate. 159

<sup>(</sup>b) WM = Weld metal.

As in the case with the 18Ni steels, the fracture-toughness testing of transweld tensile specimens of both the (250) and the (200) grade of 9Ni-4Co steels was conducted using the partial-thickness-crack (PTC) method. Only three specimens of the 9Ni-4Co(250) steel were tested because of difficulty in growing the semielliptical fatigue cracks. The specimens tested consisted of one produced from steel using the VAR-C-DOX melting technique, and two from steel produced by the Si/Al-DOX-VAR technique. The  $K_{
m IC}$  values for these specimens were between 43 and 46 ksi $\sqrt{
m in.}$  , indicating little difference in fracture toughness as a function of deoxidation technique for the 9Ni-4Co(250) grade. The fracture toughness 9Ni-4Co(200) material in plate of 0.375-inch thickness was somewhat greater in the VAR-C-DOX steel than in  $\mathrm{Si/Al} ext{-}\mathrm{DOX-VAR}$  steel, the  $\mathrm{K_{IC}}$  values for the former steel ranging from 104 to 122 ksi√in. Test results on the 0.750-inch-thick plate were similar. The apparently greater toughness of steel produced by the VAR-C-DOX melting technique resulted in excessive net stress and yielding for the specimen size and crack geometries employed, thereby leading to conservative toughness values. Table 60 summarizes the PTC fracture-toughness-test results on the 9Ni-4Co steels.

Macrosections and hardness surveys were conducted on steel for each thickness and grade. The hardness along the weldment in 9Ni-4Co(250) material was essentially constant, indicating satisfactory heat-treatment response of the filler metal. Hardness values on the 9Ni-4Co(200) grades of steel indicated a substantial spread in hardness through the thickness of the plate. The final weld pass on each side of the 9Ni-4Co(200) plate transformed to self-tempered martensite on cooling to room temperature. This resulted in a hardened area in the weld heat-affected zone. Prior weld passes had, however, been additionally tempered by the subsequent weld passes. As a consequence, softening occurred, which brought their hardness value down until it was about equal to that of the parent metal.

TABLE 60. TRANSWELD PTC FRACTURE-TOUGHNESS-TEST RESULTS FOR 9Ni-4Co STEEL

Material Description	Thickness, inch	Crack *	Crack	Crack Area in, 2			
		Length,	Depth,		σ, ksi		
		inch	inch		Gross	Net	Kic
0, 45C (Si/A1-DOX-VAR)	0.157	0.188	0.055	0,0081	118,7	120.5	43.4
•	0, 151	0.098	0.038	0.0028	166.7	167,7	46.3
0. 45C (VAR-C-DOX)	0.160	0,202	0.062	0.0098	120,1	. ~ 122, 6	45.9
0,25C (Si/A1-DOX-VAR)	0.353	0.530	0,160	0.0666	146.0	154.9	93.0
	0.329	0.478	0.115	0,0432	152.5	159.5	87.8
	0.346	0.435	0.128	0, 9437	152,9	159,8	88,5
0.25C (VAR-C-DOX)	0,359	0.713	0.208	0.116	164.2	184. 1	122.4(2)
	0.354	0.330	0.122	0.0316	196.0	202.0	104.3(a)
	0.343	0.502	0 <b>.14</b> 8	0.0583	181.3	192.2	115.2(a)
0.25C (Si/Al-DOX-VAR)	0.720	0.405	0.114	0.0362	164.3	176.3	94.1
	0.726	0.388	0.110	0. 033 ნ	163.4	174.3	88.8
	0,739	<b>0.44</b> 5	0.122	0.0428	174, 1	18Ն.6	101. g( a)
25C (VAR-C-DOX)	0.731	0.455	0.133	0.0475	179.2	196.3	107.4(a)
	0,726	0.397	0.217	0.676	173.7	198.8	103.4(a)
	0.740	0.390	0.130	0.0398	182.9	196.9	103.4 1

<sup>(</sup>a) Kic value is questionable because of excessive net stress.

#### CONCLUSIONS AND RECOMMENDATIONS

# 18Ni Maragir, Steel

- (1) The 18Ni maraging steels are subject to cracking during the reheating and forging of large-size (43-inch-diameter) ingots. Longer soaking times, higher preforging heating (at 2300 F), and slower postforging cooling lessened the tendency to crack.
- (2). It is recommended that 18Ni maraging material that is to be flame cut be overaged at 1100 F before cutting.
- (3) The preferred "finishing" temperature for 18Ni maraging forgings was found to be 1900 to 1950 F for air-melted material and 1850 to 1900 F for vacuum-arc-remelted material.
- (4) Superior toughness was exhibited by vacuum-arc-remelted as compared to airmelted material and by the (250) grade as compared to the (300) grade.
- (5) 18Ni maraging sheet finished at 1550 F exhibited properties superior to those of that finished at higher temperatures.
- (6) Optimum mechanical properties were obtained by a 1-hour solution anneal at 1500 F followed by aging for 3 hours at 925 F.
- (7) Stress-corrosion tests suggested that sensitivity decreased with decreasing strength and that vacuum-arc-melted material was superior to air-melted material.
- (8) The 18Ni maraging steels could be welded with little loss in strength and/or toughness, provided the material was re-aged after welding.

#### 9Ni-4Co Steel

- (1) Cracks were formed in the 9Ni-4Co steels during initial forging tests. The difficulty was overcome by careful surface preparation and by using a preforging temperature of 2200 F.
- (2) The preferred "finishing" temperature for 9Ni-4Co forgings was found to be 1900 to 1950 F.
- (3) The 9Ni-4Co sheet and plate was rather insensitive to "finishing" temperatures, but there appeared to be some advantage to finishing the sheet bar at 1850 F and the sheet at 1500 F.
- (4) Both grades of 9Ni-4Co steels are susceptible to stress corrosion.
- (5) The 9Ni-4Co steels could be welded satisfactorily,

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development of 18% nickel maraging steels and 9% nickel, 4% cobalt quench and temper steels. The effect of a number of processing and fabrication procedures on mechanical properties including toughness and stress corrosion was evaluated and is reported herein. None of the grades tested exhibited exceptional sensitivity to forging conditions but there was indication that the 18% nickel maraging steel possessed superior mechanical properties when reduced about 75% and finished at 1800-1850 F. Optimum results were obtained wher the 9% nickel- 4% cobalt steel with 0.45% carbon was reduced 75% but the 3% nickel -4% cobalt steel with 0.25% carbon exhibited better properties when reduced only 25%. The preferred finishing temperature for these steels was found to be 1900-1950 F. The results also suggest that in most cases vacuum-arc remelting results in some increase in fracture toughness. All of the steels proved to be rather insensitive to the rolling temperature but the greater reduction that the thinner plate underwent apparently resulted in improved toughness at least in the longitudinal direction. Weldability tests indicated that the 18% nickel maraging steel could be welded without severe degradation in heat-affected-sone mechanical properties provided the ; aterial was re-aged after welding. Welds of near 100% efficiency were produced in the 9% nickel-4% cobalt steels by quenching and tempering after welding.

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	(250) Steel	Ì					
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